

# THE CHEMICAL AGE

VOL LVI

26 APRIL 1947

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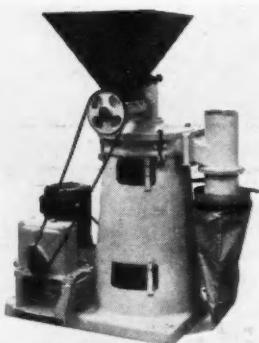
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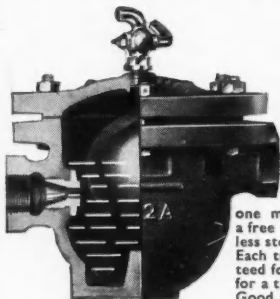
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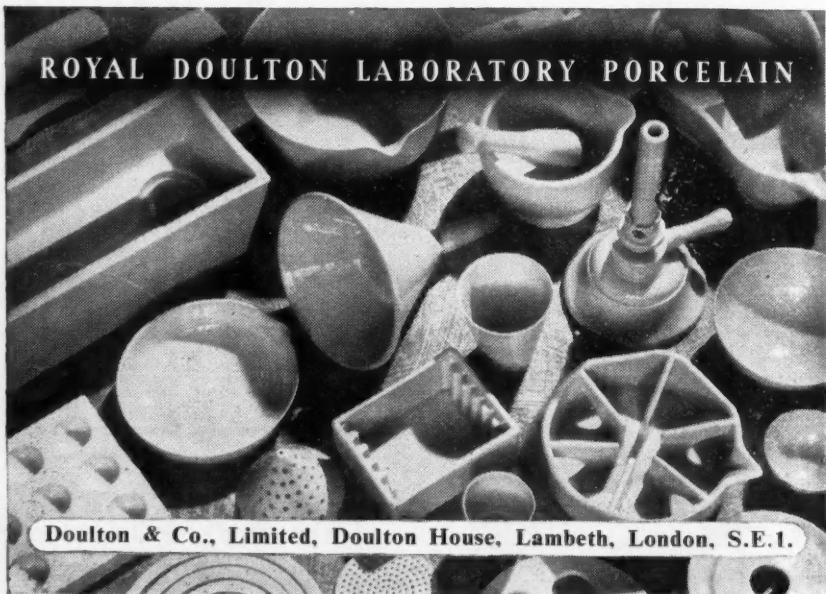
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## INDEX TO ADVERTISERS IN THIS ISSUE

	Page		Page
Advance Components Ltd. ... ..	xxxvii	Kent, George, Ltd. ... ..	545
Air Conditioning Corporation (Jeffreys) Ltd. ... ..	xl	Kent, James, Ltd. ... ..	xiii
Alcock (Peroxide) Ltd. ... ..	xxii	Kestner Evaporator & Engineering Co., Ltd. ... ..	ii
Allen, Athole G. (Stockton) Ltd. ... ..	viii	Key Engineering Co., Ltd., The ... ..	Cover ii
Allen, Edgar & Co., Ltd. ... ..	Cover ii	Laporte, B., Ltd. ... ..	xxx
Angel, H. Reeve, & Co., Ltd. ... ..	xvi	Leeds & Bradford Boiler Co., Ltd. ... ..	xlviii
Atwater & Sons Ltd. ... ..	xxii	Leigh & Sons Metal Works ... ..	xlviii
Automatic Coil Winder & Electrical Equip- ment Co., Ltd., The ... ..	xix	Leitz, E. (London), Ltd. ... ..	xxiv & xxv
Avimo Ltd. ... ..	iv	Lennox Foundry Co., Ltd. ... ..	xlviii
Baker Perkins Ltd. ... ..	vi	McCulloch Bros. & Wilson ... ..	xx
Balfour, Henry, & Co., Ltd. ... ..	xxvi	Metropolitan-Vickers Electrical Co., Ltd. ... ..	Cover iv
Basic Minerals Ltd. ... ..	xl	Mitchell, L. A., Ltd. ... ..	xxxii
Bellingham & Stanley Ltd. ... ..	xxxiv	Mullard Wireless Service Co., Ltd. ... ..	xviii
Blackwell's Metallurgical Works Ltd. ... ..	xlviii	National Enamels Ltd. ... ..	xxxiv
Boots Pure Drug Co., Ltd. ... ..	xxviii	Negretti & Zambra Ltd. ... ..	iv
Bowmans (Warrington), Ltd. ... ..	xlviii	Norman Engineering Co., Ltd. ... ..	i
British Gas Council ... ..	xxxviii	Nottingham Thermometer Co., Ltd., The ... ..	xxxvi
British Labour Pump Co., Ltd. ... ..	xlii	Oertling, L., Ltd. ... ..	Front Cover
Brough, E. A. & Co., Ltd. ... ..	xlv	Oliver Pell Control Ltd. ... ..	Cover iii
Central Office of Information ... ..	xxi	Pascall Engineering Co., Ltd., The ... ..	vii
Cooke, Troughton & Simms Ltd. ... ..	547	Perry & Hope Ltd. ... ..	548
Cruikshank, R., Ltd. ... ..	Cover ii	Pilot Engineers Ltd. ... ..	xvi
Doulton & Co., Ltd. ... ..	ii	Potter, F. W. & Soar Ltd. ... ..	xxviii
Dryden, T. ... ..	xx	Premier Filterpress Co., Ltd., The ... ..	xlviii
E.C.D. Ltd. ... ..	xxxii	Quickfit & Quartz Ltd. ... ..	xiv
Edwards, W. & Co. (London), Ltd. ... ..	xxxix	Roneo Neopost Ltd. ... ..	xlvii
Evans Adlard & Co., Ltd. ... ..	Cover iv	Shawinigan Ltd. ... ..	xxxiv
Feltham, Walter H. & Son, Ltd. ... ..	xlviii	Short & Mason Ltd. ... ..	xxiii
Foxboro-Yoxall Ltd. ... ..	v	Siebe Gorman & Co., Ltd. ... ..	xii
Gallenkamp, A. & Co., Ltd. ... ..	xvii	Spence, Peter & Sons Ltd. ... ..	xx
General Chemical & Pharmaceutical Co., Ltd., The ... ..	xlvii	Steele & Cowlshaw Ltd. ... ..	xxxvi
General Metallurgical & Chemical Ltd. ... ..	xxxvi	Steiner, Dr. L. A. ... ..	xviii
Gowlands Ltd. ... ..	xxx	Swift & Co. Pty. Ltd. ... ..	xlvii
Griffin & Tatlock Ltd. ... ..	xi	T. & T. Works Ltd. ... ..	Cover iii
Harris, Francis W. & Co., Ltd. ... ..	xliii	Thermal Syndicate Ltd., The ... ..	x
Haughton's Metallic Co., Ltd. ... ..	xlviii	Thomson, Skinner & Hamilton Ltd. ... ..	xxxv
Hearson, Charles & Co., Ltd. ... ..	ix	Towers, J. W. & Co., Ltd. ... ..	xxvii
Hull, City of, Development Committee ... ..	i	Vacuum-Physical Laboratories ... ..	xliii
Imperial Chemical Industries Ltd. ... ..	xli	Voss Instruments Ltd. ... ..	Cover iii
International Pulverisers Ltd. ... ..	Cover iii	Watford Chemical Co., Ltd., The ... ..	xxxii
Jenkins, Robert & Co., Ltd. ... ..	xlii	Whilems, C. J., Ltd. ... ..	xx
Jobling, James A. & Co., Ltd. ... ..	xiv & xxxviii	Widnes Foundry & Engineering Co., Ltd. ... ..	xxxiii
Johnson, Matthey & Co., Ltd. ... ..	xv	Wild-Barfield Electric Furnaces Ltd. ... ..	xxix



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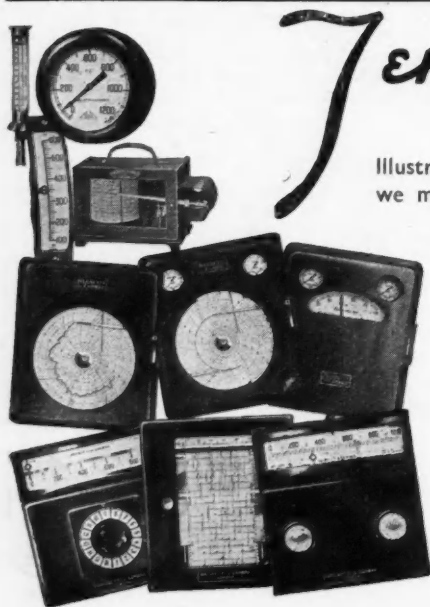
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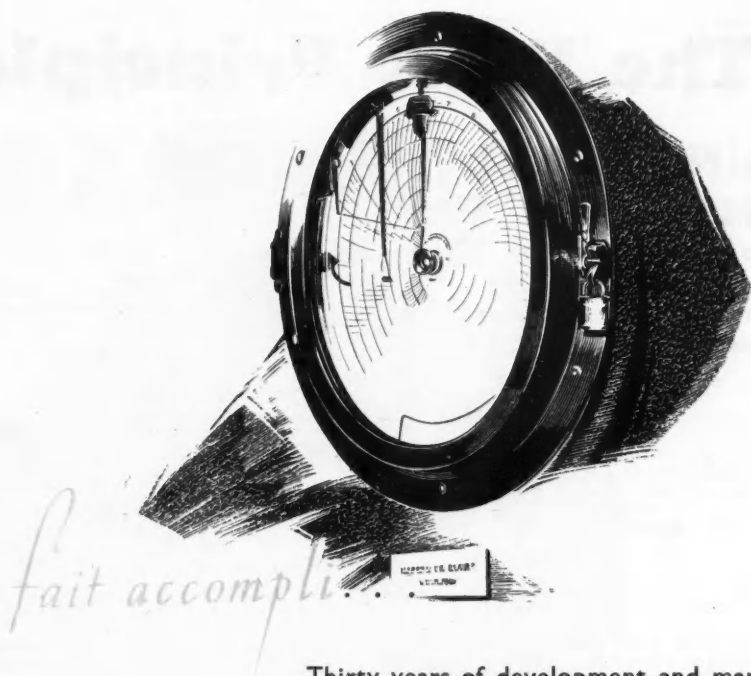
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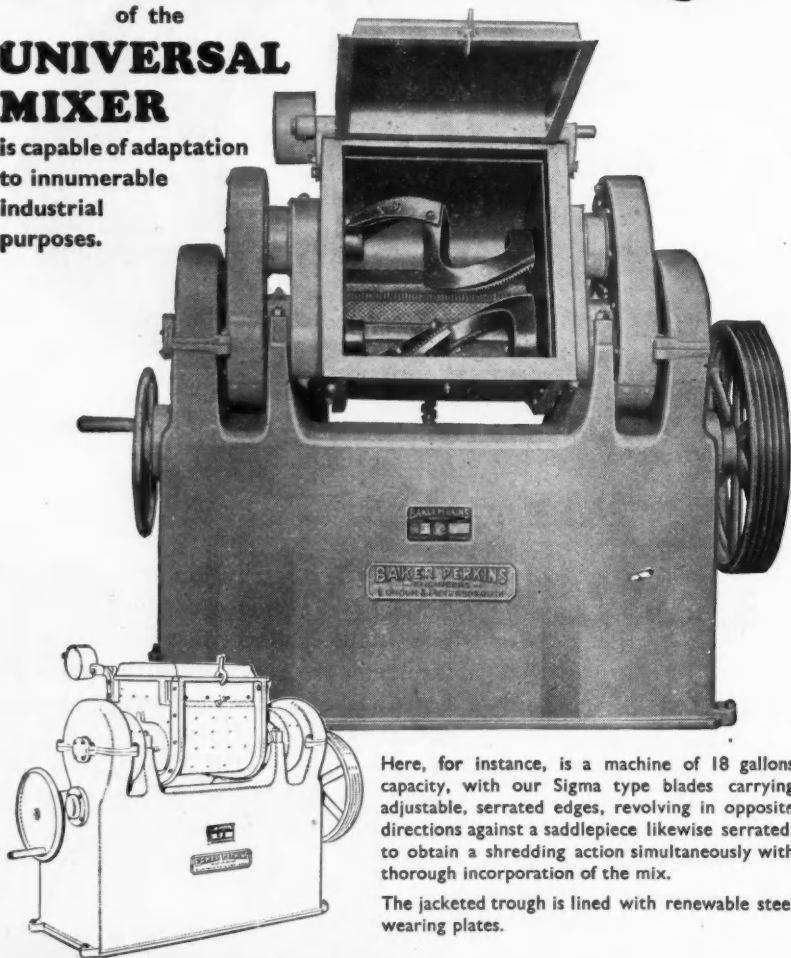
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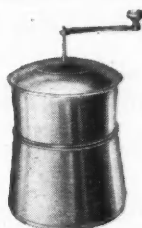
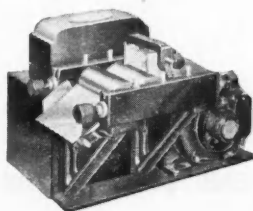
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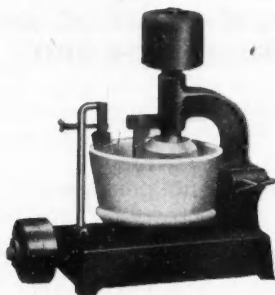
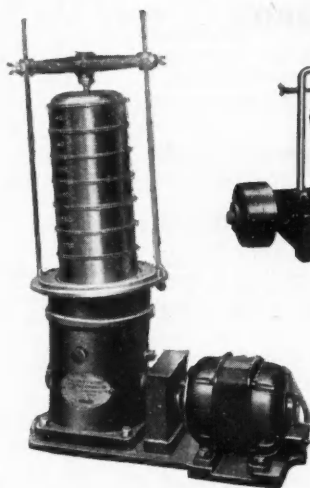
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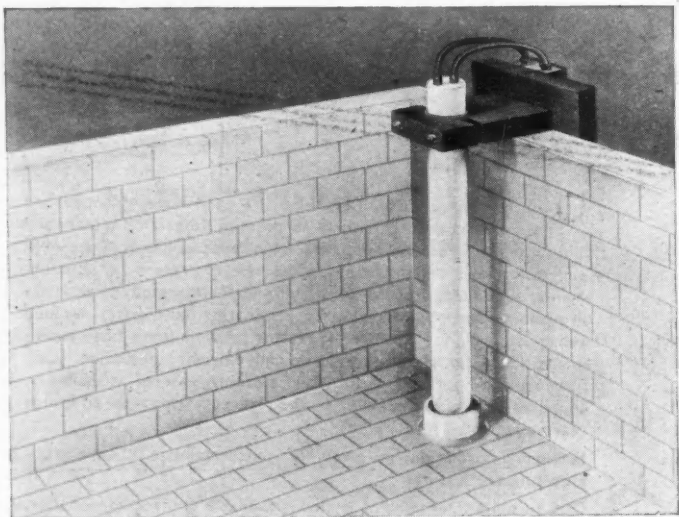
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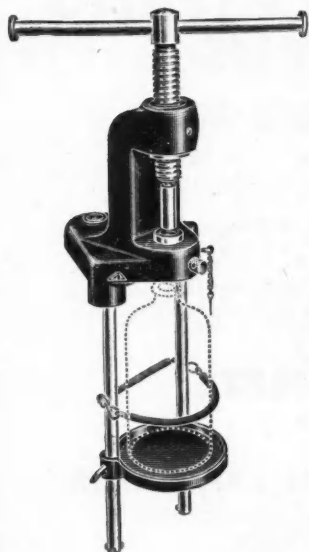
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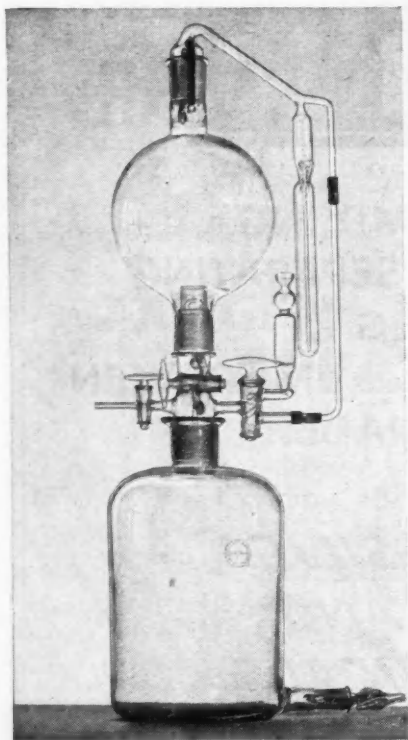
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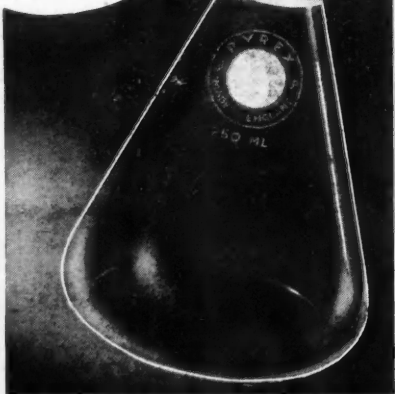
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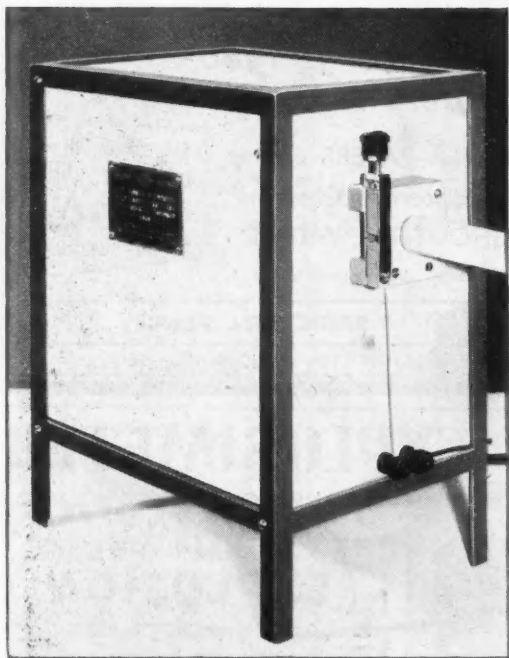
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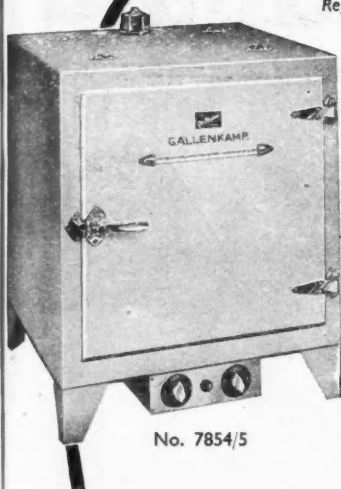
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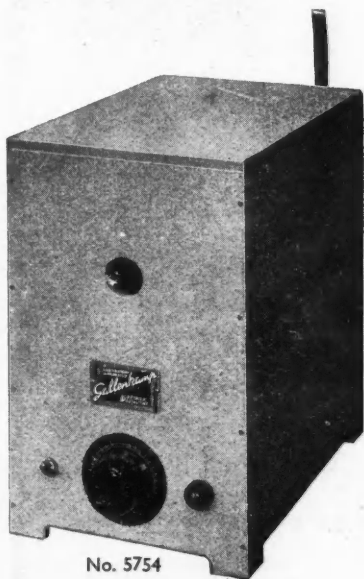
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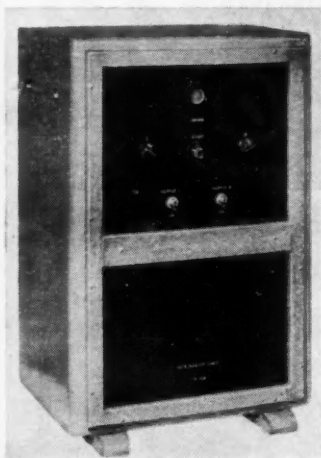
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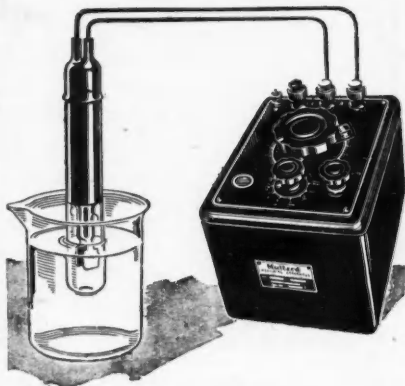
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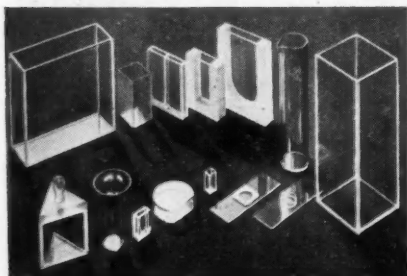
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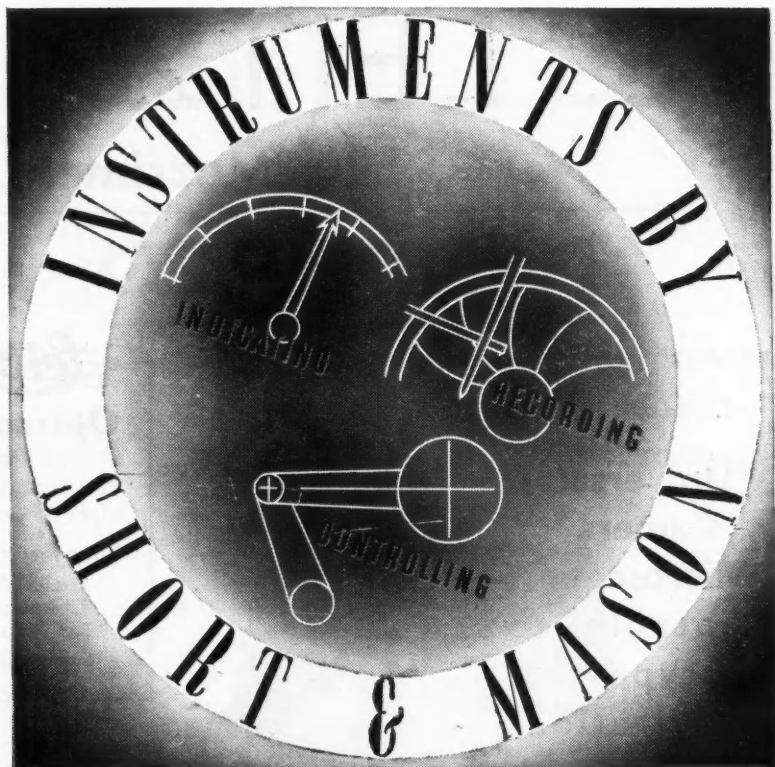
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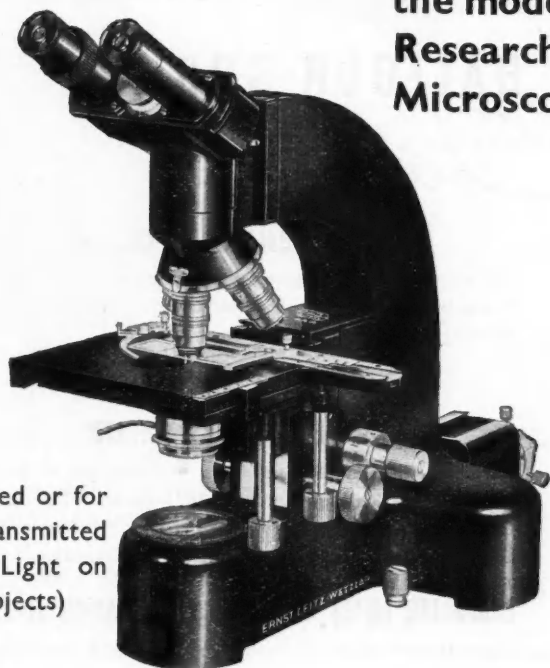
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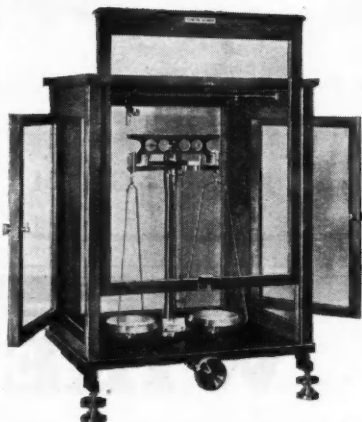
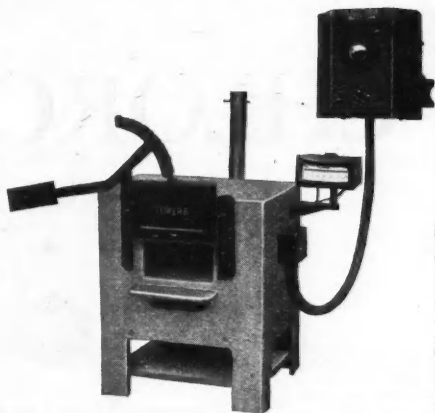
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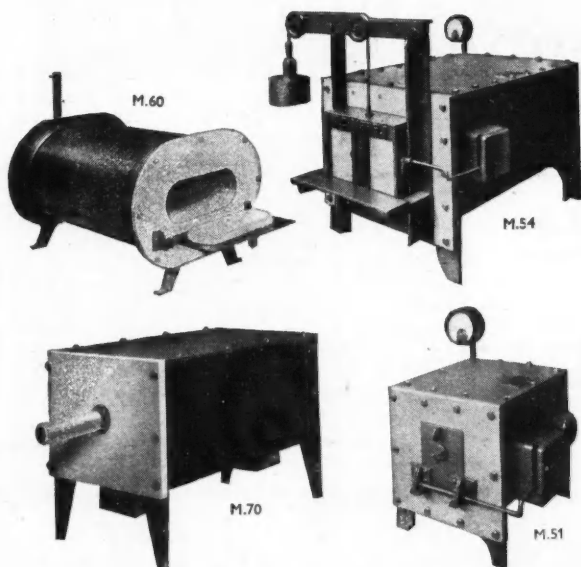
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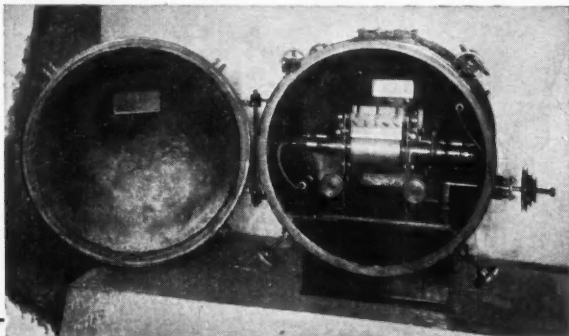
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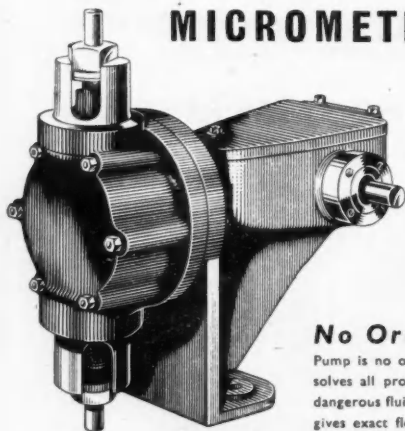
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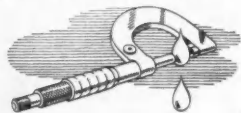


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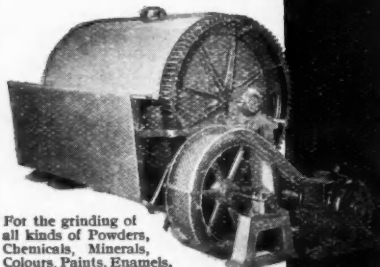
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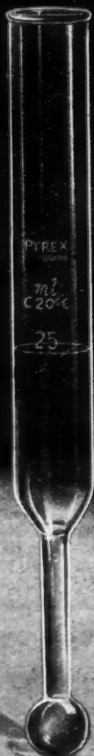
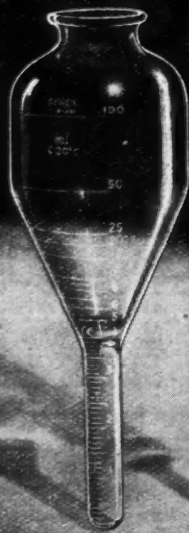
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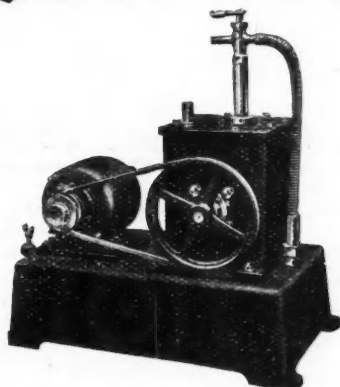
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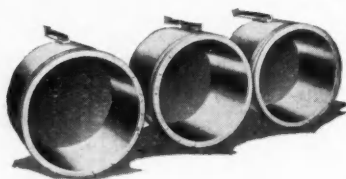
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## Chemical Engineering

THE profession of chemical engineering is among the most arduous in the world of industry. It demands many years of study and much practical application, but its rewards are commensurate with the toil involved in attaining proficiency. The president of the Institution of Chemical Engineers pointed out at the annual luncheon that if success be measured by remuneration, the chemical engineer could count himself successful, for his was among the highest-paid professions in the country. Not everyone will measure success by that yardstick; there will always be many who demand that their work shall give them the mental stimulus that accompanies the satisfaction of a worth-while job well done. By this yardstick also chemical engineering ranks among the most satisfactory of professions.

There are, however, drawbacks. In Joadian vein, the chemical engineer will ask himself at times whether there is such a thing as a chemical engineer. He will ask himself whether in fact he exists, and he will be bound to reply (to himself, for he would not dare to say it aloud): "It depends what you mean by a chemical engineer." If by a chemical engineer one means a man who combines in himself the skill and knowledge of a good chemist with those of a good mechanical engineer, we have little hesitation in declaring that, with the exception of a very few gifted individuals, there are no chemical engineers. If, however, we mean a man who possesses a fair working knowledge of chemistry and of mechanical engineering, so that he can design in broad outline the plant for an intricate chemical process, stating the dimensions of the apparatus,

the manner of their arrangement, and the broad details of the whole installation, leaving to the chemist the research work necessary to devise the process and to the engineering draftsman the details of design, then there ARE chemical engineers. The chemical engineer is the missing link between the chemist and the engineer. Mr. Geoffrey Heyworth hinted at that in his speech at the luncheon. Chemistry and engineering are each subjects demanding many years of study and practical experience and to master them both would take too large a slice out of the lives of most ordinary men. One can be mastered and the other sampled, while wisdom will grow by experience, until in the course of years the chemical engineer may approximate to the double-headed chemist-engineer that many people fondly imagine the chemical engineer to be.

The time taken for his training, even to the degree to which we have indicated, is long. Mr. Geoffrey Heyworth put it as 11 years. He is five years a-training, and to that must now be added at least another year for military service—unless the nation has the sense to decree that those who have professional qualifications can serve the country better in the works and in Government posts than in the "P.B.I." with rifle on shoulder. Having acquired a plentiful supply of book knowledge, the chemical engineer must then acquire experience in practice before he can become a successful designer. That again takes not less than five years. Thus, the chemical engineer, though perhaps destined to climb to such heights that he must pay income tax at 19s. 6d. in the £, will be some 30 years of age before he can earn

## On Other Pages

<i>Leader: Chemical Engineering</i> ...	493	<i>Chloride Determination</i> ...	518
<i>Notes and Comments:</i>		<i>Semi-Technical Chemical Plant</i> ...	519
<i>Laboratories and Instruments</i> ...	495	<i>New Use for Hydraulic Control</i> ...	527
<i>Security No Spur</i> ...	495	<i>Progress in Instrumentation</i> ...	528
<i>Production/Power</i> ...	495	<i>Electrical Measurements</i> ...	529
<i>American Explosion</i> ...	496	<i>Measuring Metallic Grains</i> ...	529
<i>Cartoon</i> ...	496	<i>Australia's Science Review</i> ...	530
<i>Texas Explosion</i> ...	497	<i>American Chemical Notebook</i> ...	531
<i>Ammonium Nitrate Precautions</i> ...	501	<i>Notes from Holland</i> ...	532
<i>France Needs Fertilisers</i> ...	502	<i>Beet Sugar Manufacture</i> ...	533
<i>Critical World Shortage</i> ...	502	<i>Welsh Steel Plan</i> ...	534
<i>Laboratory Planning</i> ...	503	<i>International Patents</i> ...	534
<i>Sub-Micro Analysis</i> ...	505	<i>Official Notices</i> ...	534
<i>The Oil Palm</i> ...	508	<i>Activated Alumina</i> ...	535
<i>Coal Conversion Plan</i> ...	508	<i>Future of German Coal-Oil Industry</i> ...	537
<i>Ceylon's Mineral Researches</i> ...	508	<i>Swedish Imports</i> ...	538
<i>Small Equipment</i> ...	509	<i>Chemist's Bookshelf</i> ...	539
<i>Laboratory Glassware</i> ...	510	<i>Parliamentary Topics</i> ...	540
<i>Radio-Chemical Laboratories</i> ...	514	<i>Next Week's Events</i> ...	540
<i>Rapid Recording</i> ...	515	<i>Overseas News Items</i> ...	541
<i>Industrial Pyrometers</i> ...	516	<i>Personal</i> ...	542
<i>Some Nottingham Thermometer</i>		<i>Home News Items</i> ...	543
<i>Apparatus</i> ...	517	<i>Commercial Intelligence</i> ...	544
		<i>Chemical Prices</i> ...	546

enough to keep a family. He is in this respect well below the level of the purely commercial staff. In the early years the life of the chemical engineer partakes of the character of the more severe monastic orders. This, of course, is not to everyone's liking.

Mr. Heyworth's organisation, Unilever, Ltd., has long been known as an undertaking which is in the van of progress. It was not surprising, therefore, to hear that he has a solution to this difficulty. It is to shorten the period of training. The solution is one that deserves to be widely considered and followed. It will commend itself especially by reason of the shortage of trained chemical engineers and of the need for increasing their numbers. It is to select graduates in chemistry or engineering when they have graduated and then to put them on the pay-roll of the undertaking. They are henceforth employees of the firm. They are left at the university to do their post-graduate course, but by arrangement with the professor the work is done especially upon problems that concern the work of the firm in question. All materials required are provided by the firm. In effect the student is thus gaining experience of the problems of his future firm while he is putting the finishing touches to his academic education. The

experiment is in too early stages for its success to be certain as yet. But it certainly seems to be in the right direction. In some other industries a similar arrangement is that of financing post-graduate scholarships in which the scholar works upon selected problems having a bearing on the industry concerned.

One of the difficulties of this procedure may be that it can only be applied by comparatively large and wealthy firms. The small organisation may not be able to afford expenditure on this scale. This is the day of the large organisation, however, and it cannot be doubted that the smaller undertakings will benefit indirectly from the supply of trained men that will be made available. The profession of chemical engineering is one that does not yet attract as many as it should do. One of the difficulties is lack of training facilities; this, we trust, will be remedied. Another is undoubtedly the length of time that must elapse before the young man earns enough to keep himself in anything like comfort. This may well be remedied by Mr. Heyworth's scheme. We believe that the day of the chemical engineer has dawned and much will be done now and in the future to make his path easier. Not everyone can learn the hard way.

## NOTES AND COMMENTS

### Laboratories and Instruments

THE planning of a laboratory, as an article elsewhere in this issue points out, is not simple. The laboratory should represent the combined ideas and efforts of experienced laboratory workers. The days are past, we hope, when the scientist was given a small, dingy dark room in which to work. His tools were given grudgingly, and "make-do" was often the order of the day. A modern laboratory with gleaming clean fittings and equipped with up-to-date instruments is a joy to the eye as well as pleasant to work in. The instruments now provided for the laboratory vary from delicate measuring instruments to beta-trons weighing hundreds of tons and costing an equally fabulous sum of money. The science of instrumentation is growing rapidly, fostered by such bodies as the Scientific Instrument Manufacturers' Association. We are pleased to help in the work of getting British instruments more widely known by the issue of such a special number as this.

### Security no Spur

IN a period of war when there is no unemployment to act as a spur to effort people still put their best legs forward and production on the highest level is, in fact, achieved, writes Sir Ernest Benn under the above heading. It is sometimes argued from that experience, he continues, that a similar freedom from the risk of unemployment should induce in time of peace, a similar willingness to work; the theory is that the desire to serve is no less sincere in peace than in war and can be encouraged, fostered and cultivated without the help of such penalties for failure as have hitherto existed, principally the fear of want. This fear as a spur to effort is looked back upon as perhaps the worst of the evils of the bad old days. But long before a Labour Government was elected in 1945, the State had assumed to itself some of the risks of poverty, want and unemployment. In the introduction to the Beveridge report it is claimed "that provision for most of the many varieties of need through interruption of earnings and other causes that may arise in modern industrial communities has already been made in Britain on a scale not surpassed and hardly rivalled in any other country in the world." So well pleased with this

state of affairs was the general public opinion that the Beveridge proposals for considerable extensions along the same lines received the support of all political parties. No social reformer, however enthusiastic, will argue that these arrangements have enhanced the effectiveness of labour or that, in consequence of them, the rate or quality of production has improved. On the contrary social reformers, of all shades of opinion, are now constrained to admit that, whatever good may have come from their endeavour to relieve the worker of the natural risks of his position, it is the fact that the product of the man-hour has been for these and other reasons, considerably reduced. All parties are at one in proclaiming the need for production and all are alarmed at the obstinate strength of the tendency in the opposite direction.

### Production/Power

TRADE Union restrictions, designed to spread the work over more men and longer periods, have usually ignored the fact that production is the only source of purchasing power. In order to safeguard the immediate position of the individual worker they have deliberately encouraged the inflationary evil of less work for more money and sacrificed the community to the supposed interests of a single class. On the outbreak of war this attitude was abandoned in favour of national service, but has now been resumed and intensified. Our major industry, coal mining, has been purged of everything to which the Miners' Union has ever objected. Royalties, profits, private ownership, capitalistic management, one after the other, have been sacrificed on the altar of "production for use and not for profit," and each concession made to the fetish has simply whetted the appetite for more, until the nation is reduced to acceptance of the pledge of a Communist that the present inadequate production will be maintained by adopting the five-day week. No less disappointing is the final proof that building labour cares nothing for the homeless and is willing to grind the faces of the poor more thoroughly than any of the old-fashioned tyrants. But even trade unionists are at last beginning to show alarm. The president of the Clerical and Administrative Workers' Union, Mr. R. E. Scouller, at the conference at Bridlington, declared: "The really

important factor is the lower output of the men employed, who appear to think they are getting their revenge on profiteering employers for generations of ill-treatment. Removal of the fear of unemployment meant that some other incentive must be found to induce men to give of their best and it had not yet been found. An enormous increase in output was easily possible and was completely necessary if we were to get back even to the pre-war rate of house building." So, through much travail, concludes Sir Ernest, we come to the conclusion that their is no spur in security, which remains as Shakespeare declared it to be—"Mortal's chiest enemy." Danger, risk, poverty, hunger, bankruptcy are the natural spurs to endeavour, security is apparently a dangerous form of dope. Of course, some measure of security is obtainable by sections at the cost of all the rest, but when, as now, all seek security there is no "rest" on whom to put the cost, and all alike are brought to the same miserable level. To call it "austerity" does not alter the ugly truth.

### American Explosion

**T**HE people of Texas City have our deepest sympathy in the catastrophe that overwhelmed them on Wednesday of last week. Not only were hundreds of men and women, idly gazing at a small fire in a ship's hold, blasted to death by

the explosion of the ship's cargo of ammonium nitrate, but many technicians, chemists and other workers in the Monsanto Company's nearby styrene plant were killed by subsequent explosions which razed the works to the ground. That, however, did not complete the chain of calamity, for oil storage tanks containing fuel oil and petrol became involved. The thick smoke from the burning oil coupled with poisonous fumes from the ammonium nitrate hindered rescue work, especially of workers trapped under the rubble of the chemical plant. That was not the last of the chain of disasters, for that same night another fertiliser ship blew up in the port. Why this happened is not known, but presumably burning oil from exploding tanks set fire to it. Details of the disaster are given on another page by our New York correspondent, while questions relating to the safety of stored ammonium nitrate are also discussed elsewhere.

### Science and Fuel

**P**ROPOSALS for a national fuel policy to ensure the fullest scientific preparation and utilisation of coal and its derivatives have been submitted to the Ministry of Fuel and Power by the National Smoke Abatement Society. Among shorter-term measures, the Society emphasises the need to:

- (a) Facilitate and promote fuel saving improvements and re-equipment in industry;
- (b) intensify and extend the already valuable work of the Ministry of Fuel and others on fuel efficiency;
- (c) require all new fuel-burning installations to receive the prior approval of a competent authority (a principle successfully adopted in other countries);
- (d) begin to implement as rapidly as possible the recommendations of the Fuel and Power Advisory Council on Domestic Fuel Policy;
- (e) begin at once to ensure that for each purpose for which fuel-energy is needed it shall be used only in the form and by the methods most scientifically and economically preferable for that purpose.

**American Scientists Coming Here.**—Dr. Robert E. Doherty, president of the Carnegie Institute of Technology, Pittsburgh, Pa., is one of the five Americans who have been invited to speak at the Centenary Celebration of the Institution of Mechanical Engineers in London this June.



**Studious One:** *Why were you so sure Bromine isn't found in Ireland?*

**Sporty One:** *Well, old Peptic told us it's never found in the free state!*



## AMERICAN EXPLOSION

### Fertiliser Ships and Styrene Plant Blown Up

ON Wednesday of last week one of the most disastrous explosions in American history, followed by great fires, occurred at Texas City, a small town of 18,000 inhabitants, about 40 miles from Houston. In an unprecedented calamitous chain of mishaps, started by a fire in the hold of a ship, two fertiliser ships, numerous oil storage tanks and a chemical factory blew up and laid waste by fire and blast a large part of Texas City. Hundreds of people were killed and thousands injured.

Eye-witness accounts of the disaster indicate that it was started soon after nine in the morning by a fire on a French ship, the *Grand Camp*, which was taking on board a cargo of ammonium nitrate fertiliser. After an attempt to extinguish the flames with steam had failed, the ship was towed out into the bay. Before it could get clear of the town, however, it blew up, killing hundreds of sightseers at the dockside and causing further explosions in a nearby chemical works belonging to the Monsanto Chemical Company. This, in turn, set fire to and exploded nearby oil tanks, causing large fires to start up all along the dock area.

Later that night there were two more explosions when another ship loaded with nitrates and sulphur, the *High Flyer*, blew up. This set off more fuel-oil and high-octane petrol tanks.

The first explosions on Wednesday morning were so violent that the concussion was felt over 100 miles away. A third of Texas City was destroyed and all other buildings lost their windows and had their doors torn from the hinges. One oil barge loaded with from 8000 to 10,000 barrels of oil is reported to have been hurled 200 to 300 ft. inland from the water.

#### Works Explosions

Most of the damage appears to have been caused by the subsequent explosions in the chemical works. These occupy a 15-acre site and are owned by the Monsanto Chemical Company. Styrene, a constituent of synthetic rubber, was manufactured there. According to a statement by a Monsanto official here, benzene and toluene were being used in one part of the plant, while high pressures and temperatures up to 1500° F. were being handled in another part. The explosion at the chemical works killed most of the technicians and workers employed at the plant, and completely destroyed the buildings.

Rescue work in the city was seriously hampered by poisonous oxide of nitrogen gases, which were apparently released by

the explosion of the ammonium nitrate in the *Grand Camp*, and thick black smoke from burning oil tanks. Fires started by the explosions and scattered burning oil raged unchecked to begin with because most of the town's fire-fighting equipment had been destroyed by the explosions, and many firemen killed or injured. It was not until fire engines from nearby towns arrived that the fires could be attended to.

#### Monsanto Statement

In an interview, Mr. Edward A. O'Neal, managing director of Monsanto Chemicals, Ltd., said that the total destruction of the Texas styrene plant and the grievous loss of so many highly skilled technicians was a very serious blow to Monsanto Chemical Company. He added, however, that, great as the catastrophe was to Monsanto's American associates, it would have no effect upon either the production or financial aspects of the British company. Mr. O'Neal said: "At the time of the disaster something like 50 per cent of the plant's output was still going to the U.S. Government's synthetic rubber programme. The actual loss in business to Monsanto will be a relatively small percentage of the American company's total sales. The long-term effect is rather difficult to forecast as, probably, the greatest overall loss will be in personnel.

"The Texas City plant, which was purchased by Monsanto Chemical Company from the War Assets Administration for just under £2,500,000 in August, 1946, had been operating at its full capacity of 50,000 tons since the end of the war. It supplied about 25 per cent of the styrene required for the Allied synthetic rubber programme. Synthetic rubber—or Buna-S—is composed of one part styrene and three parts butadiene. Styrene, when polymerised alone, becomes a glassy, transparent solid which is used to manufacture countless moulded plastic articles.

"Construction of the styrene plant, which was built for the Defence Plant Corporation and operated by Monsanto for the Rubber Reserve Company, was completed December 10, 1943. It was the first of the Government styrene plants to begin production."

Mr. Willis Cooper, who played a major part in the designing and construction of the Texas plant and is in charge of the design and construction of the new Monsanto plant which is to be built at Newport, Mon., gave this reconstruction of how he thought the disaster took place. He said: "As I understand from the news reports,

when the ship caught fire in the docks they decided that they could not extinguish the flames so they started to tow it out into the Gulf of Mexico. The only way out to the Gulf is through a very narrow channel which is very close to the Monsanto property. As the ship passed the plant it evidently exploded with sufficient force to damage some of the equipment. Although the process requires the use of benzene and toluene, it is not, in itself, dangerous when adequately controlled—as it has been for the past four years. But it would be dangerous when there was a major break in the process lines or vessels, and that is what happened here. The distillation towers were 212 ft. high, and there were 30 of them, ranging from small ones of 2 ft. in diameter to others of 11 ft. The distillation process, however, would not be the principal hazard. That would be in the gas processing — dehydrogenation,

thermal and catalytic cracking—where we handled high pressures and temperatures—up to 1500° F.

"This plant," he added, "like a large percentage of chemical operations, had a danger factor and Monsanto recognised this and took the necessary safeguards. The hazards in our plant were equivalent to those expected in an average oil refinery. The fact that the plant operated from March, 1943, until to date without major mishaps speaks for itself. Monsanto's overall safety record has been quite outstanding. Last year there were four disabling injuries per million man hours per annum compared with the American chemical industry's record of ten disabling injuries for the same number of man hours. I can judge from the pictures I have seen in the Press that there was obviously no time to take any precautions. It just went off with a bang."

## \$20,000,000 Monsanto Plant Blasted

Explosion Affects Plastics, Synthetic Rubber and Oil Industries :

Monsanto Plans to Rebuild

By LEON D. GRUBERG.

New York : Saturday.

**A** FREAK accident, caused by the explosion of a quantity of ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) aboard a freighter in the harbour, this week laid waste to the industrial port of Texas City, Texas, killing 600 people, injuring up to 1000 more, and in its wake levelled the huge \$20,000,000 Monsanto Chemical Company's styrene plant, a primary source of a basic chemical raw material in the plastics and synthetic rubber industries. The blast also wrecked several oil refineries and oil tanks in the city. Monsanto officials announced that 307 of its 500 employees are still missing and as soon as conditions permit the ruins of the company's plant will be searched by rescue squads and workers whose efforts thus far have been blocked by flames and fumes. Destruction of the plant, which company officials plan to rebuild on the same site as soon as practicable, was the result of a chain of explosions set off by the blowing up of the nitrate-laden ship, *Grand Camp*, a 7,176 ton freighter owned by the French Line and which is said to have been insured for \$600,000 (American dollars) by a British firm. Property damage from the explosion and fires will total millions of dollars.

The Monsanto Chemical Company plant represented Texas City's largest single enterprise. In addition to destruction of this plant, the Topping Plant of the Republic

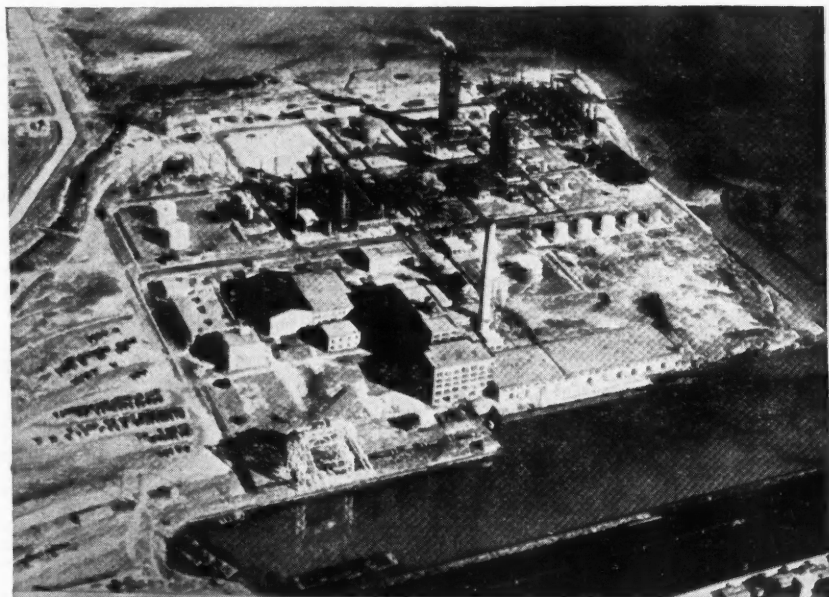
Oil Refinery Company was heavily damaged and the largest oil tank of the Humble Oil Company at the water-front was blown up. The six major industrial plants in the area were reported to represent a combined investment of \$125,000,000. Industrial activity of the city was centred in such fields as cotton, cotton bagging, sulphur, grain, chemicals, petroleum and other products.

During the war, Texas City was a major wartime industrial centre. The Monsanto plant was built by the company at a cost of \$19,000,000 for the government during the war and has been operated by Monsanto since 1942 for the production of styrene used in making synthetic rubber. One of its processes involved the cracking of propane to yield ethylene—used in hospitals as an anaesthetic. Last August, Monsanto purchased the plant from the (U.S.) War Assets Administration for \$9,550,000. The company acquired the plant to enlarge its output of polystyrene, a plastic moulding material, and recently earmarked an additional \$1,000,000 for plant expansion.

Besides its activities in synthetic rubber and polystyrene, Monsanto is a large producer of heavy chemicals, intermediates, medicinals and fine chemicals, phosphorus and phosphoric acid compounds, solvents, tar acid chemicals and other types of plastics.

Maurice Le Brozec, of Le Havre, France, a survivor of the *Grand Camp's* crew, expressed the belief that the tragic explosion resulted from combustion caused by the





**An aerial photograph of the Monsanto Chemical Company's styrene plant at Texas City, which was completely destroyed by the explosion.**

closing of the ship's hatch on its cargo of ammonium nitrate fertiliser. Normally, the hatch is left open, but because of the rainy weather of the previous days, the hatch was closed so the fertiliser wouldn't get wet. It was also reported that the *Grand Camp* carried sixteen cases of ammunition loaded at Antwerp for Venezuela, but had been overshipped and had missed port. The ammunition, according to Samuel F. Muecke, deputy collector of Customs, Galveston, Texas, was to have been transferred to another vessel at Galveston.

The Monsanto Chemical Company's installation, which bore the brunt of the damage, was completely destroyed. The styrene plant provided one-fourth of the styrene used in the country's synthetic rubber plants and accounted for one-fifth of Monsanto's total sales volume of \$100,000,000 annually. The plant had a rated productive capacity of 70,000 tons of styrene annually and was the sole source of this material for the company's production of polystyrene plastic molding materials. The company expects to produce production of polystyrene plastic moulding materials in 1947. According to officials, the explosion also demolished a polystyrene manufacturing unit adjacent to the styrene plant which would immediately cut Monsanto's output of the moulding material from

10 to 20 per cent. There was no explosion within the chemical plant or its outbuildings, it was pointed out and neither was Monsanto owner of the vessel that blew up nor did its cargo originate from any of their plants. The crumbling walls of the building started small fires which spread and ignited propane and benzol stocks. President William H. Rand of Monsanto pointed out that the plant did not explode, but caught fire, saying that the "materials we handle are flammable but not explosive."

Officials of the Union Carbide and Carbon Chemicals Corporation, which has a plant at Texas City, announced that their property suffered virtually no damage. At 3.30 a.m. Thursday, April 17, (the morning following the day of the explosion, which took place 9.12 a.m. Wednesday), Carbide and Carbon's plant was closed down as a precautionary measure. Operations at the plant will be resumed as soon as local conditions permit. Other plants in the area that were undamaged were: Pan-American Refining Company, and the tin smelter owned by the (U.S.) Reconstruction Finance Corporation. The refineries of both the Stone Oil Company and the American Liberty Company were set ablaze and the largest oil tank of the Humble Oil Company at the waterfront was blown up.

The explosions which damaged oil plants and tanks at Texas City will probably not affect the public supply of oil, especially since this is an off-season in the industry. James E. Pew of Philadelphia, an executive of the Sun Oil Company, said that other oil companies would take up the commitments of the firms whose plants were damaged.

### Explosives to Fertilisers

In an interview with this reporter, Dr. Amos Turk, instructor and lecturer in Chemistry at the College of the City of New York, said that "ammonium nitrate is usually a stable chemical when pure. It could be made impure, and therefore explosive by the presence of combustible impurities such as wood dust, rope, asphalt, or other organic matter. It could also be made impure by inefficient reclamation processes used in reconvertng ammonium nitrate from military explosives, of which it is a component. Unless checked for impurities by chemical analysis, recurrences of this type of explosion are not unlikely."

In peacetime, ammonium nitrate is used as a fertiliser and since the end of the war left large stocks of explosives containing ammonium nitrate, these are being processed and reconverted into the pure substance for use as fertiliser. Contaminated or impure ammonium nitrate is an unpredictable substance. The greatest explosion in history, prior to the atomic bomb, took place in Oppau, Germany, in 1926, when a large quantity of ammonium nitrate exploded, wiping out the entire city and killing hundreds of persons. That explosion, according to explosives experts attending a meeting of the American Chemical Society at Atlantic City, N.J., was caused by contamination of the ammonium nitrate.

The process of extracting ammonium nitrate from war explosives requires the application of a certain degree of heat. If the temperature is too high it may lead to making the compound less stable. Thus heat, as well as the possibility of contamination, tends to increase the probability of an explosion. The ammonium nitrate in its purified form comes out as a white powder. It is a colourless crystalline salt, formed by the union of ammonia and nitric acid. This is shipped in waterproof bags or in barrels. The waterproofing of the bags, the experts declared, might carry unsuspected contaminants that get into the fertiliser. Heat, such as that which came from the reported fire aboard the *Grand Camp*, might speed up the mixing of the contaminating chemicals in the bags with the fertiliser. Some have guessed that the flames must have got at the compound, which decomposes at 410 deg. F. and caused it to blow up. The experts pointed out the advisability of shipping such material in small batches rather than in one large shipload.

When ammonium nitrate becomes impure or is contaminated and explodes, it does so with about 25 per cent more violence than does TNT. Consequently if the *Grand Camp* had 10,000 tons of ammonium nitrate aboard, the explosion would have equalled 12,500 tons of TNT.

Texas City, the site of the devastating explosion, is a comparatively new industrial community—an outgrowth of two world wars. It houses six major industries, which in addition to the wrecked Monsanto plant includes the only tin smelter on the American continent, several refineries, the gigantic Union Carbon and Carbide Chemical Plant, and terminal facilities for a sea-train link with New York City.

The tin smelter, owned by the Reconstruction Finance Corporation, represents an investment of nearly \$8,000,000 and is the largest smelter in the western hemisphere. The smelter has averaged production of 3,800 tons a month. It was contracted for by the Government in February 1941 and its first output started on April 5, 1942. The purpose in building the smelter was to relieve the United States of dependence upon Europe for refined tin.

As an aftermath of the blast, and as a precautionary measure in preventive medicine, the entire city is to be sprayed with DDT from aeroplanes

### Fresh Start

On the industrial front, William Rand, president of Monsanto, announced that the company's wrecked plant at Texas City will be rebuilt on the same site. Very little of the ruined structure is expected to be salvaged and the new construction will be started as soon as it is practicable to clear away debris. The new plant will have the same production capacity as the one it is replacing.

Early reports on the insurance coverage indicated that the workmen's compensation and general liability coverage on the Monsanto Chemical Company plant was placed with the Liberty Mutual Insurance Co. of Boston. The Liberty Mutual is said to have carried a \$50,000 net line in workmen's compensation. It is understood that the Liberty Mutual's re-insurer, the American Re-Insurance Company of New York, had a \$50,000 net retention, placing the balance with Lloyd's of London. The fire and extended coverage insurance on the plant, it is reported, was with the Associated Factory Mutuals.

Insurance to be paid for loss of life and property damage may run from \$50,000,000 to \$100,000,000, according to insurance experts. The impact on the insurance companies will be softened somewhat by the division of the risks among various concerns. Of the various types of insurance, use and occupancy insurance, will be costly.

This type of insurance provides for the payment to companies forced out of their properties of an amount equal to the net profit they would have earned during the period of non-occupancy. Such insurance provides for the payment of salaries and wages of a skeleton working force, for payments on advertising and other contracts, and for various continuing expenses. Marine insurance, on the other hand, may prove costly to British underwriters.

Widows and other dependants of Monsanto

Chemical Company employees killed in the explosion will each be paid \$1,000, according to Joseph R. Mares, general manager of the company. This is in addition to benefits to be paid under workmen's compensation and group insurance life and accident policies. Payment of financial assistance to widows has already been begun by Monsanto. Injured employees of the company will receive workmen's compensation benefits plus a company contribution which will make their total compensation equal to their normal basic wage.

## Ammonia Nitrate

### Safety Precautions : Other Disasters

THE loading and transport of ammonium nitrate have assumed unusual prominence during the last few days because of the explosion which occurred on the French ship *Grand Camp* on April 16, and by reason of the two further explosions when, in the darkness before dawn, another ship, loaded with nitrates and sulphur—the *High Flyer*—suffered a similar fate.

#### No Bulk Storage

Since the Oppau explosion on September 21, 1921, in the Ammonsulfatsalpeter stores, the storage of ammonium nitrate in bulk has been avoided and, moreover, steps have invariably been taken to granulate the product to give particles of 8-16 mesh, drying to 0.2 per cent moisture, treating with 3 per cent of a suitable conditioning agent and storing in waterproof bags. The conditioning agents which have been used for the most part are kaolin and various qualities of kieselguhr. Many firms on the European continent have mixed ammonium nitrate with clay or kaolin as a barrier, and prepared granules, with a view to lessening the possibility of detonation.

The well-known I.C.I. product—Nitro-chalk—is a mixture of about 50 per cent ammonium nitrate and 50 per cent calcium carbonate. Thus, it contains about 15½ per cent nitrogen, half as ammonia and half as nitrate. The product is granulated and offered to the market in an excellent physical condition.

Plants owned by the United States Government furnish 19 to 24 per cent of the estimated nitrogen consumption and represent an investment of more than £40 millions in nine synthetic ammonia plants, having a combined capacity of 750,000 tons of fixed nitrogen per year. At the beginning of 1947 the United States nitrogen product industry was operating at near capacity. This refers to privately-owned or leased or government-controlled nitrogen fixation plants. Those remaining under ordnance ownership and control, it is gathered, were at the four arsenals, Morgan-

town, West Henderson, Texas Panhandle and Missouri. It is believed that these were being operated on behalf of the Army to make ammonia for ammonium nitrate export to occupation areas, and presumably the ammonium nitrate made at Texas Panhandle had been loaded into the ships *Grand Camp* and *High Flyer*.

That American chemists were *au fait* with the best method of loading ammonium nitrate, from the viewpoint of safety, to the ships in which it was loaded cannot be doubted. An investigation into this matter was conducted some three years ago, and a definite conclusion reached. Whether the coating of the granules with a water-repellent material, such as had been recommended, namely, paraffin wax, may have been a factor in what occurred cannot, of course, be said.

It would appear, from a first-hand eyewitness account of the explosion, that a fire started in the hull of the *Grand Camp*, and that a large number of the crew were standing around watching the efforts to subdue it when there was a terrific blast. The blast blew the hatches open, killing most of the crew, and the wreckage rained down for many minutes. Whether it was the 900 tons of ammonium nitrate which caught fire, whether it was the inflammable material which was part of the cargo, or whether it was the cases of ammunition cannot at this juncture be said.

#### Damage at Long Range

The flying debris apparently punctured oil storage tanks on the wharf, and, similarly, drums of styrene and benzol tanks at the Monsanto works some thousand yards away. The havoc grew and the conflagration gained in intensity. How precisely the second ship became involved is not known, as it was many hours afterwards when this exploded. But when one reads (*The Times* report of April 18) that a former war correspondent who flew three times over the burning city said that "in four years of reporting war he had seen no such concentrated devastation, such

utter destruction as at Texas City, except Nagasaki," one can appreciate how a succession of fires and perhaps sympathetic explosions intensified the destruction wrought.

Cargoes of inflammable material and ammonium nitrate, and cargoes of ammonium nitrate and sulphur, for which the shipping authorities are presumably alone responsible, are not necessarily the best combination of materials to be loaded to the holds of ships, even if they are kept separate.

This disaster recalls two similar incidents in our own country. One was at the works of the Explosives Loading Company, Faversham, in 1916, when all but one of rather more than a hundred employees disappeared in a gun-cotton explosion that caused damage as far away as Queenborough, in the Isle of Sheppey. The other was the Silvertown explosion in 1917, which occurred at a works on the north bank of the Thames where T.N.T. was being recrystallised by a solvent process. Ignition of the vapour took place and the resulting explosion was devastating in the extreme. Part of Silvertown was completely wiped out and other districts suffered devastation. The loss of life was serious. One of the largest gasholders in England, on the south side of the river, more than a mile away, suffered the effects of blast and repercus-

sion. It became punctured and about 7 million cu. ft. of gas were ignited, and a blaze of colossal dimensions lit the sky momentarily. But miraculously the governor attendant switched over from the damaged holder at the psychological moment and turned to another holder, and the lighting of South London continued uninterrupted.

As a precaution against epidemics, Texas City is to be sprayed from the air with DDT, and it is gratifying to think that 400 lb. of penicillin has been flown to the scene of devastation. These aids were not available in 1916-17 and, while they are undoubted advantages, it would seem that where oil or inflammable liquids are stored at docks or wharves, or adjacent to docks or wharves, fire-fighting equipment should be modernised, extended and foam for extinguishing fires should be a compulsory provision.

Profound sympathy is extended to our American friends, to the Monsanto company and to the relatives of all those who have lost their lives, and not less so to those who have been injured. It is understood that an investigation is to be made into the circumstances of the disaster, and the report of the findings will be awaited with interest, for no one can afford to ignore the import of the precautionary measures which presumably will be issued relative to the loading of such cargoes.

### France Needs Fertilisers

**F**RANCE'S urgent need of greatly increased supplies of chemical fertilisers is underlined by a survey made by A. Marquis for the information of the *Centre de Perfectionnement Technique* (*Chim. et Ind.*, 1946, 56) in which he reveals the extent to which French husbandry has been starved of chemical stimulants since the beginning of the war. The shortage of phosphates has been most marked. Totals for the five-year period 1940-41 to 1944-45 were: nitrogen 725,000 tons, phosphoric acid 468,000 tons, and potash 880,000. What was formerly a three-year supply of nitrogen and potash has thus been made to last for five years, and in the same period the amount of phosphoric acid available was equivalent formerly to one year's requirements, notwithstanding that even before the war 75 per cent of cultivated land in France was deficient of phosphates.

The urgency of the problem of enlarging supplies of chemical fertilisers has been stressed in the past before the Academy of Agriculture by A. Demlon, who drew attention to the low agricultural returns, a recent example of which was a reduction of 20-30 per cent in the 1944 sugar beet yield.

### Critical World Shortage

**T**HE International Emergency Food Council announces recommendations for distribution of the world supply of nitrogenous fertilisers will be continued in the fertiliser year commencing July 1, 1947. As from that date, however, all other fertiliser materials will be withdrawn from the list of reserved commodities on which recommendations are made. Previously, new supplies of phosphate rock becoming available after January 1, 1947, had been withdrawn from the reserved list. The present action of the Council takes potash and soluble phosphates off this list after July 1, 1947. At the same time, the Council, through the Committee on Fertilisers, reported that a critical world shortage of nitrogenous fertilisers will continue through 1947-48; and it is estimated that the world shortage for the present year is at least 761,000 tons of nitrogen (equivalent to 3,805,000 metric tons of ammonium sulphate). The Committee found that world production of soluble phosphates will be approximately in balance with world requirements in 1947-48, though some areas such as Austria and Germany may still find difficulty in meeting requirements. The position of potash is less clear.

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# Laboratory Planning

## "Combined Operation" for Expert and Architect

**P**LANNING of a laboratory is not simple. If it is to achieve results making for maximum efficiency it calls for an intricate "combined operation" on the part of both laboratory expert and architect. Only the laboratory expert understands the ultimate requirements; only the architect knows how those requirements can be met.

In an exhaustive survey of the subject in a series of articles in recent issues of the *Indian Journal of Scientific and Industrial Research*, Mr. G. P. Contractor, of the National Metallurgical Laboratory, Council of Scientific and Industrial Research, gives detailed guidance in the matter.

In his introduction, Mr. Contractor emphasises that in laboratory planning it is desirable that the important features of construction and equipment should represent the combined ideas and efforts of those who have years of experience as laboratory workers. It is equally essential, he says, that both laboratory workers and architect should work together to analyse the requirements of the project in order that every need be satisfied as completely as possible.

Efficiency and convenience of operation coupled with the required degree of flexibility and adaptation to future needs should always be borne in mind. The size of the rooms must be carefully planned with respect to the requirements for benches and aisles, especially for the chemical laboratories, and for illumination.

### Safety Measures

Properly planned laboratories should, according to Mr. Contractor, have, among other things, the greatest possible working area for a given floor space; a minimum number of places where dirt and dust can collect; non-corrosive hardware and fixtures; adequate services and outlets; easily cleaned well lighted hoods; suitable general illumination; easily cleaned durable walls and floors; and safe construction and equipment. The possibility of fire and explosion can be lessened by proper attention to venting of all pipe chases in both walls and floors, using a colour code for easy and positive determination of service lines, suitably located easily shut off valves, regular testing of high pressure equipments, adherence to code requirements in electrical installations and taking precautionary measures for the storage of easily inflammable material. In case of fire there should be readily available fire extinguishers in working condition, quick acting showers or fire blankets and suitable alarm devices.

Injury due to mechanical causes can be minimised by providing adequate lighting, especially in hoods, halls and stairways and at machines by removing obstructions in passage ways; and by suitable location and construction of doors. First-aid equipment in usable condition should be readily available, and each laboratory should have posted conspicuously information as to the locations of master switches and valves.

### Co-operative Effort

After emphasising that successful planning of a laboratory is dependent upon close co-operation between architects, engineers and laboratory workers later to use it, Mr. Contractor points out that planning does not merely consist in the number and sizes of rooms and their inter-relation for convenience and service but should also refer to the position of electrical conduits, piping and ventilating systems, as well as lighting. To these, he says, may be added consideration of the correlation of chemical units with architectural units, such as location of vents, windows, piping, etc., in respect to the design of the building.

The writer quotes the concept that a laboratory, to be utilised to the best advantage, should be built round the equipment; and he illustrates it with, among other examples, the layout of spectrographic laboratories carried out around the apparatus and other requisites.

The articles emphasise that it is poor policy to erect a building to meet only present needs, making no provision for future additions.

Chemical fume hoods, in Mr. Contractor's opinion, constitute a very important part of the equipment of a laboratory. Because of the corrosive, poisonous and obnoxious fumes, excessive heat and the ever present danger of fire, he points out, the hoods should necessarily be built of materials that will effectively resist these agencies. The materials usually employed are white or mottled glazed brick, enamel tile and other products which go under the trade names of impregnated "Transite" board, "Sindanyo" asbestos board, "Labite" sheet, "Alberene" stone, "Standard Composition" stone, etc. The working surface is built of soapstone, fireclay tile or similar acid and fire-resisting material. It is also often built of ordinary cement concrete. Furthermore, hoods should be scientifically designed and properly proportioned not only to take care of the correct amount of air uniformly distributed over the hood opening, but also to exhaust completely and

effectively all of the light and heavy fumes from the entire chamber.

Various classifications of hoods, which merge more or less into each other have been made, such as individual and general or side wall hoods, open and closed, up and down draught, natural and forced draught, etc. The usual types are either of open-front or sliding sash type. The sash is glazed with laminated tempered glass or preferably with wire-glass for safety. In case of fire, one need only close the sash and shut off the emergency valve on the outside panel of the hood. Where the bulk of routine analysis for control of plant operations is required to be carried out, the long open type hooded work bench is preferred to the sash type hooded work bench. It is important to bear in mind that the primary object of a hood is to enable a worker to carry on with ease and comfort a given reaction while at the same time observing and controlling the process. It is reckoned that nine-tenths of the reactions evolving objectionable fumes carried out in a chemical laboratory can undoubtedly be carried out in an open hood. A survey of the best modern laboratories indicates that for more advanced work and for general industry and research, an increased use of open hoods is being made.

### Room for Storage

Working benches provided with essential services to ensure convenience, which he describes as the most important fitting in a laboratory, are fully dealt with by the writer. He adds that facilities for storage of chemicals and glassware should be provided in the work bench; but points out that it is not desirable to store chemicals in cupboards beneath a bench, as an accident involving spillage of liquids may damage the contents of the cupboard. Some planners are in favour of open work benches, the storage cabinets and shelves being conveniently located behind the worker. When this arrangement is fol-

lowed, the cabinet work can form a useful partition between benches. It also gives a measure of privacy to workers on individual benches without being completely shut off from the workers on adjacent benches.

The articles conclude with a few general hints to laboratory planners:

All laboratories should be provided with at least one safety overhead water shower as well as with modern equipment for fighting small fires. "Foaming" and carbon tetrachloride extinguishers in useable condition should be kept at easily accessible points.

Raw linseed oil applied hot in a thin layer is still one of the best protections for bench tops when they get dingy. The oil may be diluted by an equal volume of turpentine.

### Electric Points

It is desirable to provide main circuits of sufficient size to allow for 50 to 75 per cent overload at the outlets originally installed. A 10 ampere capacity receptacle mounted on a standard box is a very convenient outlet for alternating current.

One electrical outlet for each 3 ft. of bench front is usually reckoned to be sufficient.

For distilled water-supply, block tin lined bronze piping should be used. Aluminium pipes have also been used in some laboratories. Block tin pipe is the best material for the purpose.

Separate stores outside the main building should be provided for inflammable substances and acids.

Laboratories should be provided with a junk room. Many a piece of half worn-out apparatus has been resurrected from a junk room and put to good use.

Balances and other delicate measuring instruments should be insulated to protect them from vibrations. This could be effected by providing independent piers running clear through floors for individual balances and instruments.

## High Vacuum Evaporation

### A Means to Fine Deposits

WHEREVER the deposition of molecules of a given material is required the setting up of high vacuum conditions greatly facilitates the process. This is the basis of the Metropolitan-Vickers Electrical Company's high vacuum evaporation plant, which, while it is intended primarily for the deposition of silica on specimens for use in an electron microscope, is also suitable for the deposition of gold or other metals in connection with the shadow-casting technique. Other applications include small-scale general or experimental work on evaporated films and use as a straight-

forward high vacuum pumping plant. The plant consists essentially of a glass bell-jar evacuated by a "Metrovac" type 03B oil-condensation pump which is backed by a "Metrovac" type DRI rotary pump. Heater terminals are provided inside the bell-jar and are connected to a low voltage transformer, the primary input to which can be varied. Vacuum valves are provided to permit continuous operation of the oil-condensation pump. The plant is totally enclosed and all electrical controls are located on the front panel.

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# Sub-Micro Analysis

## Valuable Applications in a Widening Field

by CECIL L. WILSON, M.Sc., Ph.D. F.R.I.C.

**B**EFORE, and during, the war, I was often puzzled to explain to non-scientific friends just exactly how microchemistry was of use. After a careful explanation of the generally recognised applications, I still had the uncomfortable feeling that all the information gathered by my friends was that the applications were few, limited and not particularly important. Nowadays the problem is simpler. One need only remark that microchemistry made the atomic bomb possible.

This, although quite satisfying to my non-scientific friends, is, of course, almost as inaccurate as their previous impressions. The methods employed in determining the properties of plutonium and neptunium are so specialised and so out of the run of ordinary microchemistry that few microchemists have had occasion to use them or to become familiar with them.

### "Microgram Chemistry"

This being so, it is possible that some account of techniques which are now normally referred to as "sub-micro" may prove of interest. By generally accepted standards, semi-micro methods deal with weights of material of the order of millilitres, while microchemistry in the strict sense is "milligram" chemistry, in that samples usually weigh one to ten milligrams. The volumes of solution used may roughly be assessed as lying between 20 cu. mm. and 0.5 ml. These two branches are the small-scale fields generally familiar. The corresponding figures in sub-micro analysis lie round several micrograms ( $\mu\text{g.}$ ) and 1 cu. mm. As a consequence, sub-micro methods are often more precisely referred to as microgram chemistry.

Because of the problems involved in reduction to this scale, the development of sub-micro methods has not been as rapid as the development of micro methods. In its early days it was only applied to highly specialised problems and the methods were diversified and not widespread. Biochemical investigations frequently called for methods which could deal with these small amounts, and it is not surprising to find that it is in biochemical literature that most of the earlier techniques were described.

### Minute Samples

One of the classic developments was the design of the Rehberg burette. The investigation which had been undertaken by Rehberg<sup>1</sup> involved the determination of blood-ammonia on quantities of blood of the order of 1 ml. Such an amount of blood

contains about 1 to  $2\mu\text{g.}$  ammonia, so that titration with a normal solution would give a titration figure of 0.1 cu. mm. Obviously it might be possible to tackle such a prob-

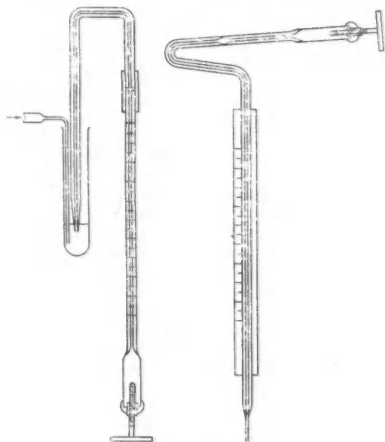


Fig. 1.

Fig. 2.

lem by using very dilute solutions in amounts then easily measurable. However, Rehberg showed in his paper that, speaking generally, small amounts of acid or alkali cannot be titrated exactly, using diluted solutions of low normality. In a comparative calculation, where 10 cu. mm. of  $N$  acid in a volume  $x$  are titrated with 0.1  $N$  alkali, the error if  $x = 100$  mls. is 10 per cent, whereas if  $x$  is 1 ml. the error is only 0.2 per cent. It follows from this that the titration solution should have as small a volume as possible, and in the example quoted above, where  $x = 1$  ml., the amount of alkali added should be of the order of 0.1 ml. This raises the further difficulty that it must be possible to add this 0.1 ml. with an accuracy of at least 1 per cent.

Prior to Rehberg's work, the usual type of microburette held a volume of 2 ml., and the greatest accuracy that could be expected is probably 2 cu. mm.

### Mercury Indicator

Rehberg's problem, then, was to design a burette capable of being read to 0.1 cu. mm. The burette (Fig. 1) consisted of a vertical thick walled capillary of bore 0.8 to 0.9 mm., giving a volume of 0.1 ml. for

a length of 15-20 cm. The calibration of the tube was in mm., and these were evaluated by determining the length of a weighed drop of mercury at various points. The lower end of the tube was widened to form a mercury reservoir, and this was closed off by a plug holding a screw. By turning the screw, mercury could be pushed up to fill the graduated tube completely. At the top the tube was joined to a capillary tip of similar tubing, bent by a double right angle, to dip into the titration fluid, and drawn out somewhat at the tip. The tip is kept immersed in the liquid in the titration vessel during titration, and liquid is expelled by moving the burette screw. The volume expelled is read from the change in the level of the mercury meniscus.

The titration volumes used by Rehberg were of the order of 1 ml., and with his conditions he was able to determine ammonia with an accuracy of 0.019  $\mu$ g. A similar type of burette was used by Linderström-Lang and Holter.<sup>2</sup>

The principal disadvantage of burettes of this type arose from the fact that many of the solutions to be used in them attacked mercury. This was overcome by Kirk,<sup>3</sup> who separated the mercury and the liquid in the burette by an air space. Because of compression of the air this gave rise to a lag in expulsion of liquid when the operating screw was turned; but if titration is carried out slowly, particularly at the end point, this lag offers no great inconvenience.

### Greater Accuracy

In conjunction with such a burette (Fig. 2) Kirk used as titration vessel a microscope slide with or without a small depression in its surface. Stirring of the titration fluid, which Rehberg achieved by a stream of gas bubbles, was by means of a glass thread operated by an electromagnetic device. Titration of amounts of fluid of the order of 0.01 to 0.2 ml. was possible using this apparatus together with other ingenious devices described in the same paper, and an accuracy of  $\pm 0.2$  to 0.3 per cent on simple titration is claimed. Naturally this figure may be somewhat larger if more complicated procedures such as precipitation and solution are involved.

An accuracy was achieved, using these drop scale titrations, of  $\pm 0.5$  per cent in the determination of 0.5 to 12  $\mu$ g. of calcium.<sup>4</sup> In the determination of nitrogen<sup>5</sup> by a sub-micro Kjeldahl method, amounts from 4 to 15  $\mu$ g. were determined with a mean accuracy of 1 per cent. The same accuracy in the determination of ammonia<sup>6</sup> on amounts from 1.5 to 8  $\mu$ g. by a diffusion method was also recorded. These results, of general interest, illustrate the methods which were applied by Kirk and his co-workers to a wide range of biochemical determinations. Similar types of investigation

were undertaken by Sobel and his co-workers, and a comparison made of the micro and sub-micro methods<sup>7</sup> shows how normal methods may be adapted to this scale.

These few examples chosen from a wide range, while illustrating the type of work which can be attempted on the biological side, must of necessity omit reference to many such estimations. For example, it is impossible to do more than refer to the outstanding work of Linderström-Lang and Holter or the extremely interesting apparatus devised by Wigglesworth.<sup>8</sup>

### Pioneer Workers

Turning to the inorganic side of sub-micro work, the ground may be said to have been broken in a classic series of researches by Benedetti-Pichler and his co-workers. These techniques are also fully described in Benedetti-Pilcher's well-known textbook.<sup>9</sup>

The earlier papers<sup>10</sup> are primarily concerned with the qualitative analysis of microgram quantities of material such as pigments in paintings. This led to an extension of the work to inorganic qualitative analysis in general. Thus, in one paper the complete analysis of a particle of Wood's alloy 1  $\mu$ g. in mass is described, which enabled not only a qualitative, but in the case of some metals a semi-quantitative, estimate of the constituents to be made.

All operations are carried out under a low-power microscope using manipulators. Separations are performed in micro centrifuge cones of special design, and confirmatory tests are applied to drops on a specially designed condenser rod. As little as 0.001  $\mu$ g., of a constituent can often be identified with certainty. Since the tiny drops of solution (0.01 to 0.001 cu. mm.) dealt with would evaporate almost instantaneously the various operations are performed in a moist chamber which has a damp atmosphere maintained in it.

Later the methods were extended to volumetric analysis.<sup>11</sup> The micromanipulation technique was still employed. It was possible to manipulate burettes with a total capacity of 0.05 cu. mm. Liquid is expelled into the titration fluid, which is held in the narrow end of the tapered tube, by using a levelling bulb balanced against the surface forces at the extremely fine tip of the burette. Removal of the burette tip from the titration fluid, by forming a fresh meniscus, stops flow of the liquid from the burette tip. The accuracy of titrations on the macro scale could be closely paralleled in sub-micro titrations in such determinations as acid-alkali titrations or the determination of chloride argentometrically.

From the published account of researches on trans-uranic elements<sup>12</sup> it is clear that



the work of Benedetti-Pichler has been largely drawn on. The diagrams of the apparatus used and the descriptions of the work are by no means as full and informative as one could wish. However, anyone familiar with Benedetti-Pichler's work will note immediately the resemblance between his apparatus and that illustrated for volumetric work and for the study of precipitation reactions under the microscope.

### Quartz Fibre Balance

The major advance here seems to be the development of a quartz fibre balance of amazing sensitivity, enabling work to be extended into the gravimetric sphere. Two balances are described. The first, which is merely a very sensitive Salvioni balance (Fig. 3) has a sensitivity of  $0.02 \mu\text{g.}$ , a weighing range of  $20 \mu\text{g.}$ , and a capacity of  $10.5 \text{ mg.}$  The second (Fig. 4), which has a counterpoised quartz fibre beam on a torsion fibre, has the same sensitivity coupled with a weighing range of  $300 \mu\text{g.}$ , and a capacity of  $25 \text{ mg.}$  Thus, with relatively heavy vessels, up to  $25 \text{ mg.}$  weight, it was possible to determine several hundred micrograms with an accuracy of well below 1 per cent; and even  $1 \mu\text{g.}$  could be determined with an accuracy of 2 per cent. Roughly speaking, if one transfers these figures to those of ordinary chemistry, it is as if one had a balance capable of taking a load of  $100 \text{ g.}$  having a weighing range of over  $1 \text{ g.}$  and a precision of  $0.1 \text{ mg.}$  This one would regard as quite a satisfactory instrument by ordinary standards, and it must be regarded as an outstanding achievement when reduced by a factor of 5000.

In the work on plutonium, the first avail-

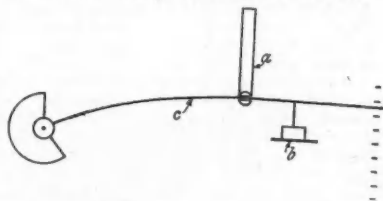


Fig. 3: a, loading stop; b, pan stirrup; c, quartz fibre  $0.004 \text{ in.}$  diameter.

able sample was  $2 \mu\text{g.}$ , and a certain amount of chemical work was carried out on this, using volumes of solution ranging from  $100$  to  $0.01 \text{ cu. mm.}$  and weights of sample as low as  $0.1 \mu\text{g.}$  Precision of  $0.5 \text{ per cent}$  was obtained in this work. Later, several hundred micrograms of plutonium were produced and made available for further investigation. Various compounds of the element were prepared and analysed with sufficient accuracy to enable their formulae to be determined with certainty.

Many properties of these compounds,

such as solubility, were investigated, and an examination of methods of separation indicated processes which would enable plutonium to be extracted in full-scale work. Although less neptunium was available than plutonium, similar applications have enabled

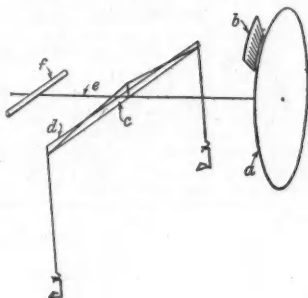


Fig. 4: a, calibrated dial; b, vernier; c, beam; d, index fibre projected by optical system; e, quartz fibre  $0.001 \text{ diameter}$ ; f, quartz bow.

the chemistry of this element to be investigated in some detail.

### Wider Use Soon

Subsequent events may focus the spotlight on sub-micro work as applied to atomic research, and there may be some tendency to feel that it has received publicity which it does not fully merit. The extraordinary efficiency of sub-micro work has, however, been displayed effectively for the first time in this work. Seaborg notes that following on the chemical determinations on microgram quantities of plutonium, the processes were scaled up directly to use in the Harford plant by a factor of  $10^{10}$ , which he remarks must surely be the greatest scale-up factor ever attempted. To use the word "attempted" also, is somewhat misleading, since the "attempt" was highly successful, and the performance of the process was much better than could have been anticipated.

It is to be hoped that full details of the manipulative technique will be published shortly. Although it is unlikely that such a spectacular performance will ever be repeated, it is clear that there is considerable scope for application, and no doubt for the extension of sub-micro investigations, both in the inorganic and in the biochemical field. It is quite clear, too, that the apparatus required for such work is not so expensive or so difficult to produce that sub-micro methods would be outside the range of the ordinary laboratory. Nowadays, routine semi-micro work is replacing or supplementing macro work in many

laboratories with microchemistry to fall back on as an emergency measure. The time is probably not too far distant when the ordinary laboratory will regard sub-micro methods as being in no way more unusual than it regards micro methods to-day.

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## The Oil Palm

### Progress in the East Indies

**A**N interesting article on the culture of the oil palm (*Elaeis guineensis*) appears in *Oleagineux*, 1947, 2, 1-10, by M. Ferrand, Research Director of the I.R.H.O. (Inst. Recherches Huiles Oleagineux) in Paris. It contains some references to the African oil palm but is mainly concerned with cultivation in Sumatra and Malaysia, where remarkable progress has been made in recent years. In Sumatra the first plantations were made in 1910 by Hallet, but in Malaysia its introduction dates back to 1875. It might be said indeed that the first beginnings on the east coast of Sumatra were really made in 1848; but most of these very early starts were abandoned.

It was not until 1920-24 that a really scientific study was made of the cultural conditions most suitable in the East Indies. An early discovery was the injurious effect of leaving the ground open, and a search for suitable covering crops or perennial plants was soon begun. The next important research was connected with the use of fertilisers, also the still more important matter of variety and selection. The methods used to-day, introduced in 1928-30, are described in some detail, including a reference to the well-known work of M. P. Michaux, "Economie des sols de plantation d'Hevea et d'Elaeis," Paris, 1936. High yields of palm kernels and palm fruit are now obtained, and an extensive programme of research for further improvement of yields in French Africa has been arranged by the I.R.H.O.

## Coal Conversion Plan

### £30 Million for Pittsburg Project

**T**HE conversion annually of some six million tons of coal into petrol, diesel and fuel oil, industrial alcohol and heating gas is the immediate aim of a co-operative scheme about to be launched by two of the largest fuel companies in America, the Pittsburg Consolidation Coal Company and the Standard Oil Company, of New Jersey. More than £30 million is earmarked for the gasifying and liquefying plant which it is estimated will produce each year 96,000 million cu. ft. of gas, 114 million gallons of petrol, 14 million gallons of diesel and fuel oil, and 8 million gallons of alcohol, the essential constituent of synthetic rubber, plastics and many other commodities in keen demand. The experimental plant is about to be erected near Pittsburg near the source of much of America's coal.

The process to be used involves successively the crushing of coal to fine particles, the generation from it of carbon monoxide and hydrogen, the combining of these to form liquid and gaseous hydrocarbons, and then their separation. After carbon dioxide—generated with the carbon monoxide and hydrogen—is removed from the gaseous hydrocarbons there will remain a high peat gas, which will be stored in gas-holders, ready for consumption. The liquid hydrocarbons, treated in a fractioning tower, will yield petrol, alcohol, diesel oil, and a small amount of heavy oils.

## Ceylon's Mineral Researches

**A** COMPREHENSIVE geological survey of Ceylon's mineral resources, not hitherto fully explored, is planned, it is officially announced.

Preliminary surveys of the island have disclosed large plumbago (graphite) resources, easily extractable deposits of an ore of titanium (ilmenite) deposits of iron ore and prolific gem fields; in addition to workable mineral deposits of china clay, glass sands, mica, cement materials and building stones, silica and other refractories, soda and alkali salts.

Before the war the gem fields and plumbago mines were the only mineral resources worked to any great extent. During the war plumbago output was greatly increased to meet the Allies' needs, and lack of imports necessitated the working of the local deposits of china clay, glass sands, mica, cement materials, etc.

The war, with its scarcities, fostered the process of industrialisation; what the Sinhalese could not import they tried to make; and with the establishment of the Department of Commerce and Industries in 1940 considerable progress was made.

## Small Equipment

### For Heating, Stirring and Weighing

**P**ORTABLE electric hot plates are virtually an essential for many purposes in the laboratory and the fitting of a thermostatic control, such as the "Simmerstat" made by J. W. Towers and Co., Ltd., of Widnes, makes the hot plate as easily controlled as a bunsen burner and without the fire risk. This form of control operates by switching the electric current on and off at predetermined intervals, giving a full range heat control from zero to maximum without waste of current. The contacts are operated by a snap action switch and are capable of operating millions of times.

The "Simmerstat" control is attached to the 3-pin plug forming a compact unit, and being removed from the hot plate is safe and unaffected by spillage on the plate. The hot plate is 5 inches diameter, has sloping sides to make it drip-proof, and is fitted with a

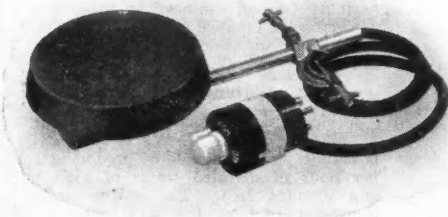
polished heat reflector on the underside. The 400 watt long-life embedded type element suffices for most general laboratory purposes. The surface temperature reaches 350 deg. C. at maximum heat.

Of almost equally wide application in most laboratories is some form of electric stirrer, of which the Towers Universal laboratory stirrer is a good example. This has a  $1/75$ th h.p. universal A.C./D.C. motor, which is mounted with the speed control in a ventilated stainless steel case. It is supported by a  $\frac{1}{2}$  inch diameter stainless steel tube which protects the electric cable. The speed control covers practically the whole range, up to 2,000 r.p.m. A chuck is provided for gripping various sizes of stirrer; and a  $\frac{3}{4}$  in. pulley which may be used for driving any other small apparatus.

Another effective application of the "Simmerstat" control is in the new Towers 10 in. seamless copper waterbath, enabling the temperature to be controlled at any required level. The heating element is of the immersion type, 1500 watt capacity, and has an automatic cut-out to prevent damage should the waterbath run dry.



This 10-inch copper water bath (above) by J. W. Towers & Co., is fitted with portable hot plate with simmerstat heat control (below)



(Above, left). A compact laboratory stirrer by the same firm with speed control in stainless steel case

# Trends in Laboratory Glassware

## Wider Use of Precision-Bore Tubing

**T**HE year has been a difficult one in the field of laboratory glassware, as it has been in many other industries, and there has been a tendency by most firms to concentrate on their well-established lines. New articles of a general nature are consequently few, but it is interesting to observe the trend of design in the application of precision-bore tubing, now becoming available in increasing quantities to which attention is given at the close of this article.

Among the apparatus of general application is the Towers Fractionating Unit (Fig. 1), designed by Gilson and Consterdine. This is suitable for research laboratories and is

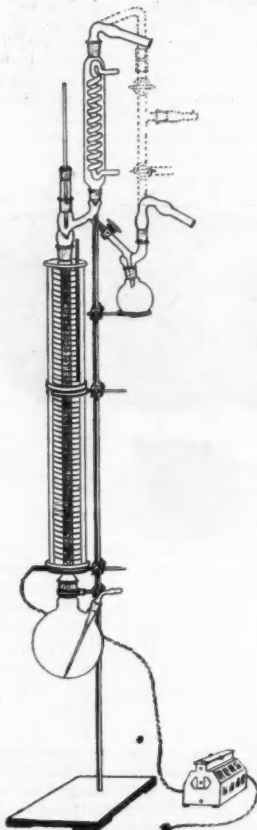


Fig. 1.

adapted for either normal pressure or vacuum fractionation. It is constructed in Pyrex glass and is fitted with standard ground joints throughout. The 24 in. column is filled with single-turn glass helices, and

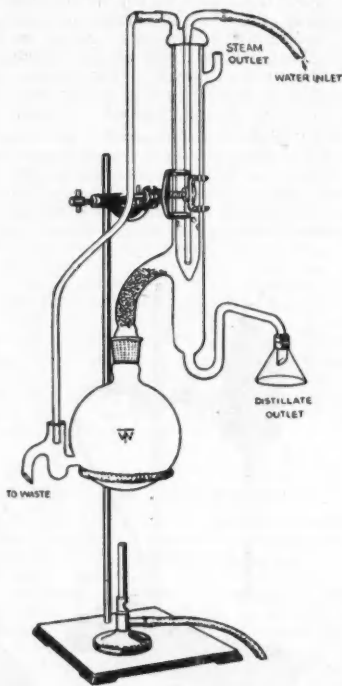


Fig. 2.

surrounded by a double walled glass heating jacket, rheostat controlled, allowing observation of the column; a total condensation variable take-off stillhead is fitted and the apparatus is supplied complete with 500 and 1,000 ml. distillation flasks and 250 and 500 ml. receivers.

### Water Distillation

A simple and robust distilled water outfit, also by Messrs. J. W. Towers, (Fig. 2) is claimed to be capable of producing 1 litre per hour of distilled water of high purity and pyrogen free. The splash baffle, an important part of the still, consists of a wide tube packed with small pieces of glass

tubing, and should prove very efficient. Otherwise, the still follows well established principles, and is suitable for use in small laboratories, dispensaries, etc. It is compact and all parts are interchangeable and readily replaced from stock.

From Messrs. Townson and Mercer, Ltd. comes the reaction flask (Fig. 3) of 6 litre capacity and made from Hysil glass, which

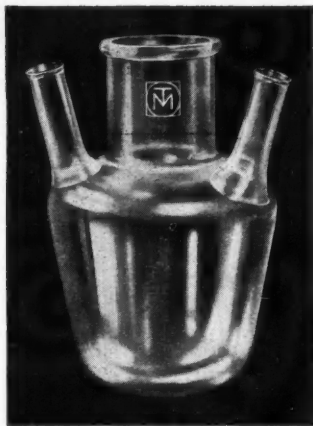


Fig. 3.

represents a departure from the conventional shape; the wide neck permits entry of a stirrer of ample dimensions, and the shape makes for easy arrangement of side-necks for delivery tubes, thermometer pocket, etc. which can be nicely positioned in relation to the stirrer. The main neck is also large enough for the hand to enter for cleaning purposes. This matter of ease of cleansing and other operations is one to which sufficient attention is not always paid, and which would repay something in the nature of research. There is little doubt that, notwithstanding the marked progress there has been in design, further critical study of small equipment will not be wasted.

#### Good Design

Micro-Kjeldahl apparatus is a fertile field for designers, but one of the neatest which has come to our notice is that depicted in Fig. 4. Designed by Roy Markham and manufactured in Phoenix glass by Messrs. F. W. Flaig and Sons, it seems to cover all the requirements of such a design, combining as it does steam-trap, still, splash-head, condenser, and caustic soda funnel, the steam-trap forming both the heater and the automatic emptying device for the still. The apparatus is supported by a single clamp

and requires only a distillation flask, provided with a clipped off tube in the stopper, for the steam generator.

Fig. 5 illustrates a group of the Ogal series of optical glass cells manufactured by The Tintometer, Ltd. This firm has successfully adopted the policy of concentrating on standard lines with a steady improvement in the supply position. Cells are being made in Hysil glass as well as the usual softer glass, and a very high degree of optical precision is maintained, matching the craftsmanship which alone enables this type of all-glass cell to be produced. They are making a number of cells suitable for continental comparators and other sources.

In sintered glass filters there has again been a concentration on standard lines, the more recent developments being shown by the adoption of standard ground joints to Buchner funnels, etc., as shown in Fig. 6, (A. Gallenkamp and Co.) and the Smith Filter Beaker, (Fig. 7) included in the B.T.L. series. Of 1½ litre capacity, this filter has the great advantage of stability, compared with that of a large Buchner funnel perched on top of a filter flask, and any capacity of receiver can be attached by a rubber lead.

#### Precision-Bore Tubing

Many interesting developments are likely to occur in apparatus design in the use of precision-bore tubing, now being produced in increasing quantities by Messrs. Chance Bros. and James A. Jobling and Co. The former for their Veridia tubing use their Hysil glass, and shrink selected length of tubing on to accurately formed mandrels. Sizes available range from 0.38 mm. to 30.00 mm., and on the standard 12 in. lengths a tolerance on bore diameter  $\pm .01$  mm. is

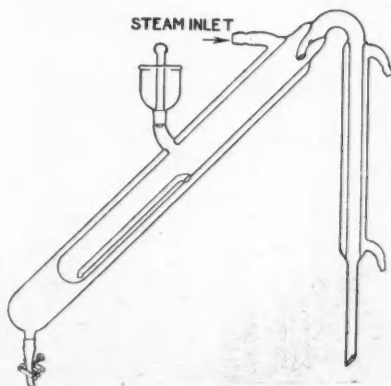


Fig. 4.

claimed. Lengths up to 48 in. can be supplied in some sizes, but here a slightly greater tolerance is required. By this process also tubes of square or hexagonal bore, or having a contoured, tapered, or stepped formation can be produced. Capillary tubing of .065 mm. bore has been made to special order.

Messrs. Jobling produce their Pyrex brand precision-bore tubing by a patented semi-continuous process, whereby selected lengths of tubing are drawn down over a short mandrel, and lengths of 6 ft. or more are handled. The tolerance on bore diameter is .05 mm., well within most requirements, and sizes are available from 3.0 mm. to 30.0 mm.

#### Tubing Technique

The uses to which the tubing may be put, come under two main headings: (1) the use of the accurately dimensioned tubing as such in the construction of apparatus; (2) the use of the syringe principle. Under (1)

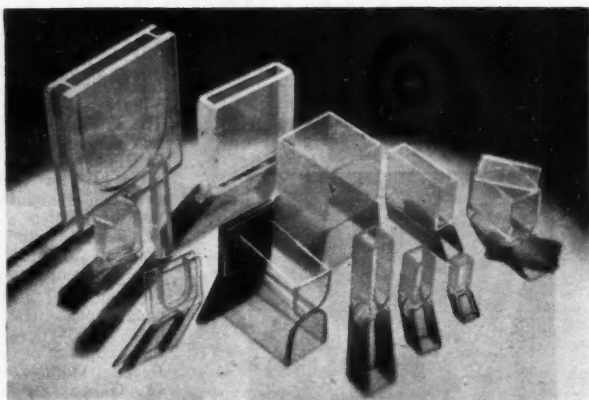


Fig. 5.

come viscometers of the B.S. U-tube form, or the straight forms adopted by the British Cotton Industry Research Association<sup>1</sup>; burettes, straight pipettes, Westergren pipettes, which permit in some instances the application of a non-calibrated scale; fractionating columns of the type provided with a closely fitting inner member of helical or moulded bulb form; specialised apparatus, such as the activated charcoal testing tube

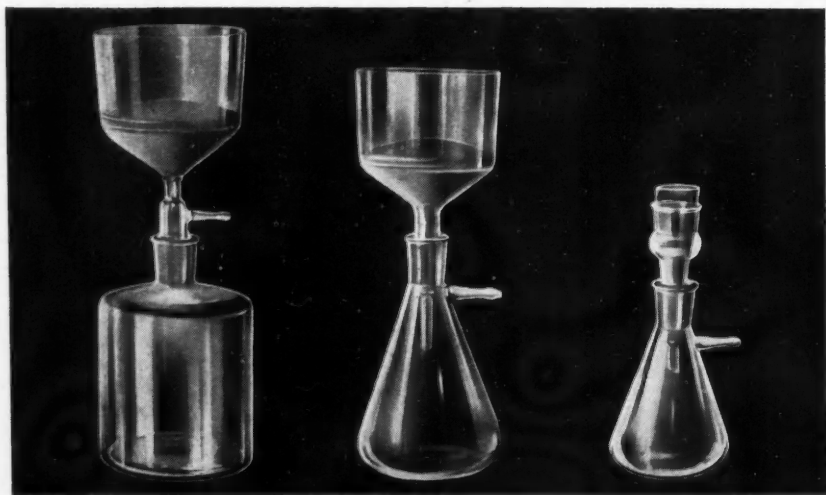


Fig. 6.

illustrated in Fig. 8. In this a 20 ml. sample of the charcoal occupies a standard length of the tube, whose bore is 20 mm. diam. It is supported on a No. 00 sintered glass disc, and an inner tube carrying a similar disc rests on top of the sample.

Much of the precision bore tubing made in this country goes to the manufacture of hypodermic syringes, for which the bore is generally sufficiently accurate for use without lapping before fitting the plunger.

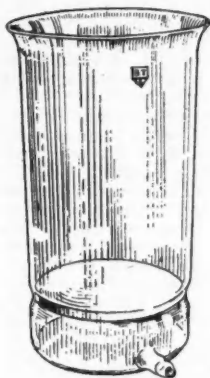


Fig. 7.

These syringes have been adapted for chemical use, both as adjustable automatic pipettes<sup>1</sup>, and, with a micrometer screw fitting, as micro-burettes or pipettes. Both of these use the hypodermic syringe in its original form; but an application in which it is used only as a pump is also in use. In this straight pipette, made by W. G. Flaig and Sons, it was essential to preclude any possibility of contact with a dangerous liquid. The addition of the pump, slightly wetted with paraffin liquid, enabled this to be done, with a possible loss of accuracy, as the pipette was not free-flowing. However, an operator continually using such a pipette, should be able to adopt a technique which will give him the accuracy he requires. An alternative construction is also employed, in which the plunger is raised, while the finger is held over the small side tube, this being released, as in normal pipette practice, when adjusting and making a delivery.

#### Automatic Pipette

Reproducible accuracy, using the syringe principle for a pipette, can only be obtained by employing glass-to-glass stops. An automatic pipette on these lines was described recently in *THE CHEMICAL AGE*.<sup>2</sup> This is an instrument which can be attached to an aspirator or other reservoir and will deliver

measured quantities of liquids with a considerable degree of accuracy and reproducibility. One is a further pipette employing the syringe principle and glass-to-glass stops, and was designed by V. Stott and I. H. Hadfield of the N.P.L. (B.P.584,841). The two stops shown on the barrel are accurately ground perpendicular to axis, and the distance between them measures the volume to be delivered. The technique of using the pipette is simple, and as the volume delivered depends only on the bore of the tube and the length of stroke, wetting of the walls, delivery time, etc. are discounted, and a reproducible accuracy of .001 is claimed.

#### New Bornkessel Burner

Messrs. Chance Bros. have recently redesigned one of their range of burners. This is a burner of large capacity, having a maximum gas consumption in excess of 50 cu. ft. per hour. With its two nose pieces and 9 jets, screwed conveniently into the heavy iron base, it is adjustable for all sizes of flame. The burner is adjustable for both angle and height, and jets can be changed without turning out the flame. The gas tube and swivel are brought closely against the line of the body to cause least inconvenience when joining awkward side-arms.

Messrs. Safety Products, Ltd., announce that they are now manufacturing goggles for glass working fitted with the new "Neoden" glass (Chance ON18). This is particularly designed to absorb sodium glare, also most of the injurious infra-red radiation associated



Fig. 8.

with working the hard glasses in an oxygen flame. These glasses thus combine the properties of didymium glass in absorbing the sodium yellow, with a further protection against infra-red. The colour transmitted is a slightly greenish-blue and does not interfere with the judgment of the lampworker.

<sup>1</sup> Shirley Institute Memoir II, 1936.

<sup>2</sup> Ayling, B.J. Exp. Path., 1924, p. 354.

<sup>3</sup> I.C.P. Smith, *Chem. Age*, Oct. 5, 1946.



# RADIO-CHEMICAL LABORATORIES

## EQUIPMENT AND HAZARDS

**T**HE rapid development of radio chemistry and its widening applications are posing new problems in laboratory furnishing to satisfy the primary need to safeguard all engaged in the laboratories, as well as the delicate equipment employed. Wherever radioactive substances are in use the urgency of providing complete protection from the harmful, sometimes deadly, emissions is well recognised, but determining the character of such protective devices is as yet far from being an exact science. Much of what has been done so far is still more or less of a tentative character and radio-chemical laboratory furnishing will almost certainly have to be re-designed many times as the level on which such work alters to conform with progressive advances in technique.

Some of the most representative examples of what has so far been done to make radio chemistry nearly as safe as other branches of science are to be found in the laboratories of the U.S.A., and an interesting survey of these is provided by a recent issue of the *American Chemical and Engineering News*.

### Degrees of Danger

The importance of the radiation hazard—our American contemporary points out—depends upon both the quantity of radioactive substance as measured by disintegration rate and on the quality of the radiation—whether alpha, beta or gamma, regardless of energy range. Since alpha rays are very easily absorbed, by a few centimetres of air or the wall of a vessel, attention is chiefly directed to the more penetrating beta and gamma rays, and in these distinction must be drawn between slightly active, moderately active and highly active samples. Although a complex of factors have also to be considered, techniques, it has been found, can be defined in three categories of radioactive intensity: (a) the microcurie level, covering activities up to 1 millicurie; (b) the millicurie level, ranging from 1 to about 500 millicuries; and (c) the multicurie level, covering activities greater than about 0.5 curie.

It is characteristic of radiochemistry that operations at the higher levels always entail associated operations—such as measurement—of lower levels and it is good practice to provide separate laboratories for operations at different activity levels and to take increasing care that the low level laboratory is not contaminated.

Operations with beta and gamma emitters at levels of radioactivity in the microcurie

range are not usually very different from most ordinary chemical operations, and can for the most part be carried out in an ordinary well-equipped laboratory. To be sure, there are new, characteristic techniques peculiar to radiochemistry experimentation, but these are reflected for the most part in minor rather than major items of equipment; similarly, serious health hazards are associated with radioactivities even at this low level, but they can be circumvented without elaborate revisions in standard laboratory design.

An exception relates to counting and other radiometric equipment. Sensitive instruments of the sort required for precise radiochemical measurements are best located in a constant-temperature room whose walls provide shielding against radiation from sources outside. The latter is especially desirable if work with sources in the millicurie region or higher is in progress in the neighbourhood. Counting rooms at Clinton Laboratories are built with 2-ft. thick concrete walls, and have air-conditioning equipment to hold the temperature close to 70°F. Care and vigilance are necessary to prevent contamination of counting rooms by quantities of radioactive materials too minute to constitute a health hazard but ample to interfere with precise measurements.

Other necessary items are a "contamination counter," or suitable substitute, for checking upon the radiochemical cleanliness of glassware and other items, and a portable radiation meter for detecting dangerous levels of radiation and ascertaining tolerance exposures. All working in the neighbourhood of the laboratory should wear pocket radiation meters, which should be read daily.

### Protective Shields

The chief requirement in chemical operations is that sources of even a few hundredths of a millicurie are never brought into close proximity with the body tissues, which can usually be avoided by use of properly designed tongs and other mechanical devices. Disposal of radioactive wastes on the microcurie level is usually not a difficult problem, since it is often permissible to use ordinary drains.

Operations at the intermediate level of activity are characterised by the necessity for shielding between the source and the operator. Two distinct techniques have been developed; one constitutes "close" shielding around individual pieces of equipment and is applicable to the less penetra-



ting radiations (beta and X-ray); the other comprises the use of general shielding and is necessary when gamma radiation is encountered.

The general shielding method involves the erection of an absorbing wall between the operator and the apparatus containing active material, manipulations being carried out with suitable devices over, around or through the barrier.

One such arrangement employs a well lighted hood having walls and floor of lead 3 in. thick with stainless steel sheet. The front wall is 6 in. thick. By raising the whole 6 ft. or more above floor level an operator standing on the floor of the laboratory is shielded from sources within the hood. General visibility is provided by a tilted mirror placed above and at the back of the hood.

When close shielding techniques are used, as with beta sources at millicurie level, it is most convenient to build a heavy-walled

jacket to each piece of equipment. The shielding jackets may be of transparent plastic, so that visibility is not sacrificed.

### Other Risks

With emitters of purely alpha radiation such as plutonium, the hazard is principally one of extreme toxicity and extreme precautions must be taken against the ingestion or inhalation of even minute quantities of the material. In plutonium work, all chemical operations should be performed inside well-ventilated hoods and a dust-free atmosphere must be maintained, to achieve which the whole laboratory should be under forced ventilation and separated by air lock from the rest of the building. Positive-pressure masks should be provided for extraordinary hazards, especially for cleaning up spilled material, and entering workers should change their clothing.

## Rapid Recording

### U.S. "Split Second" Pyrometer

**F**OR applications where a fast-changing temperature must be recorded or controlled with "split-second" speed—or where temperatures from many points should be recorded on one chart in such rapid succession that trends show up clearly, and data from hitherto unrecorded points can be included—Speedomax type G pyrometers made by the Leeds and Northrup Company, of Philadelphia, have abundant sensitivity and "across-the-chart" speed.

In experimental laboratories, pilot plants and production departments, single-point recorders of this type follow a key temperature so swiftly and uninterruptedly they detect significant fluctuations in a fraction of a second, providing an unusually detailed operating record.

### Continuous Data

In many industries multiple-point instruments are bringing to a central point the data about temperatures throughout an entire process. Because they run through 16 points in as little as a minute, one instrument can record on a single chart all important temperatures at the unusual speed which many processes require. Temperature facts about a process can be complete, conveniently usable and easy to compare.

Where temperatures from a number of points should be recorded as a check on process uniformity, with one point serving as the basis for automatic control, a multiple-

point recording controller can regulate temperature for an interval, pause briefly to record one or more of the other temperatures, then get back to the control point so quickly that process temperature has not had time to change significantly.

### Readily Adaptable

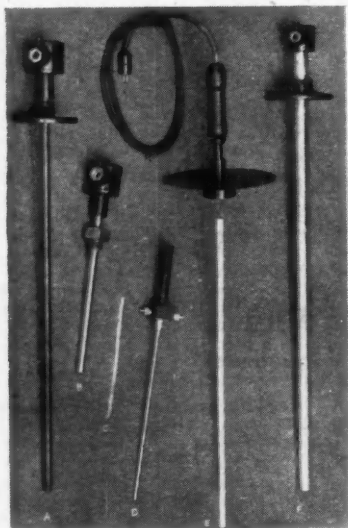
Speedomax type G is a strip-chart (Model S) pyrometer. It is available for use either with thermocouples or Rayotubes. It indicates and records, and can be supplied to operate any of the full line of the firm's signals and controls. Within all practical limits, it can accommodate any external resistance. It can be supplied for any temperature range for which other of the firm's recorders can be supplied, as well as for very narrow ranges, and for remarkably fast "across-the-chart" speeds. Instruments with standard ranges provide an accuracy of adjustment of one-third per cent of the range.

Speedomax type G can be installed almost any distance from couple or Rayotube. It is remarkably free from effects of vibration, shock and stray fields. Usually it is less affected by dirt and dust. Because its case dimensions are identical with those of the Model S Micromax, instruments of both types can be interchanged, so that a panel-layout can be rearranged for maximum effectiveness with little effort.

## Industrial Pyrometers

By the Notts. Thermometer Co.

**M**EASUREMENT of temperatures enters into nearly all industrial operations at one stage or another. To the chemical industry this factor applies with even

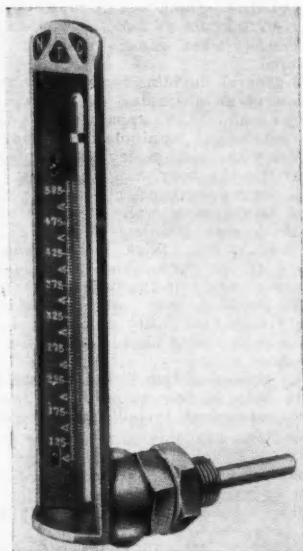


**Thermo-couples for varied purposes and use in conjunction with thermo-electric indicators and recorders**

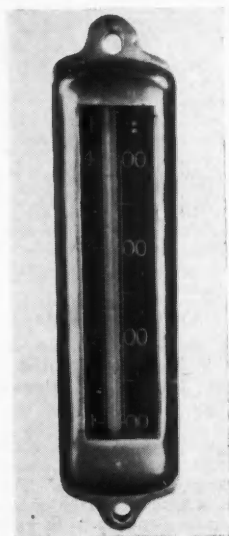
greater frequency than in most others and the electrical pyrometer generally provides the most convenient means of measurement.

### High Temperature Recordings

The thermo-electric pyrometers manufactured by the Nottingham Thermometer Co., Ltd., are designed for recording up to 1400 deg. C., and within the range manufactured by the company are indicating and recording pyrometers, controllers and potentiometers of wall and portable types, and surface temperature pyrometers for reading temperatures of sheets, billets, hot plates, revolving rolls and press platens. Simplicity of design without sacrificing accuracy of readings is the keynote of this range of instruments and they are constructed to give enduring service under severe conditions. A one year's guarantee of workmanship and material testifies to their dependability.

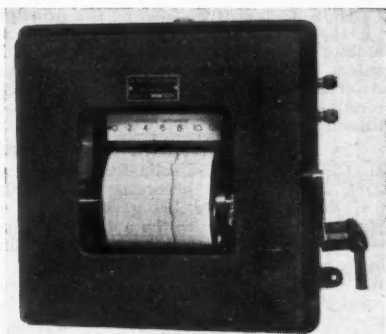
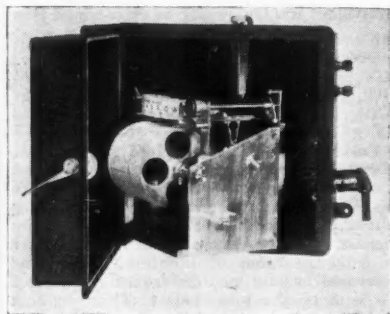


**Diesel engine exhaust thermometer**



**Oven thermometer in chromium-plated fitting in standard sizes, 8½ ins. and 10½ ins.**

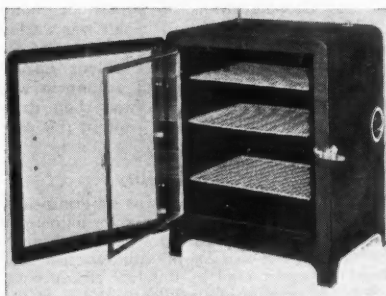
## Some Nottingham Thermometer Company Apparatus



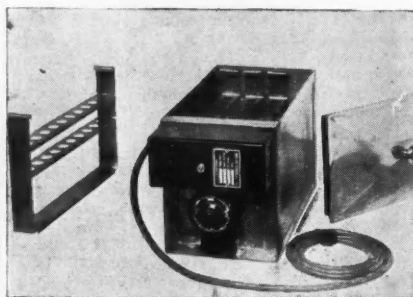
A millivoltmeter to provide a continuous record of temperature from day to day or, when a cut-in switch is on, an additional dot and dash record. The mechanism and rotating chart are shown in the left-hand picture



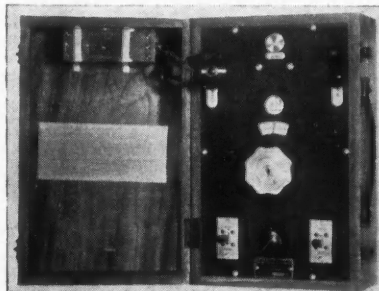
Compactly designed Gerber centrifuge



Electrically operated incubator with inner glass door, to control at any set temperature required



Constant temperature bath to hold 40 test tubes in four racks at 37.5 deg. C. Gas operated models are also available



Portable potentiometric pyrometer giving accurate measurement of temperatures from minus 200 deg. C. to plus 1600 deg. C. or Fahrenheit equivalent

## Chloride Determination by Mercurous Iodate

by K. AVALIANI\*

**T**HIS method is founded on the principle that the addition of mercurous iodate to a chloride solution gives soluble iodate equivalent to the chloride originally present. After filtration the iodate in solution is determined volumetrically by means of sodium thiosulphate.

The reaction is  $\text{Hg}_2(\text{IO}_3)_2 + 2\text{Cl}^- \rightleftharpoons \text{Hg}_2\text{Cl}_2 + 2\text{IO}_3^-$ .—The solubility in water at 25°C. of  $\text{Hg}_2\text{Cl}_2$  is according to Landolt,  $0.47 \times 10^{-4}$  g. per 100 g. and that of  $\text{Hg}_2(\text{IO}_3)_2$ , according to the author's results, is  $0.2 \times 10^{-4}$  mol. per litre; hence the solubility products are  $5.3 \times 10^{-19}$  and  $2.56 \times 10^{-14}$  respectively, and the equilibrium constant of the reaction is  $(2.56 \times 10^{-14}) / (5.3 \times 10^{-19})$  or 48300. It is concluded, therefore, that the reaction proceeds very nearly quantitatively (99.998 per cent) from left to right.

The reagent,  $\text{Hg}_2(\text{IO}_3)_2$ , was prepared by double decomposition of potassium iodate and mercurous nitrate. The salt was washed with water until successive portions of the wash water gave the same colour on the addition of a few drops of sulphuric acid and potassium iodide. It was then dried over  $\text{H}_2\text{SO}_4$  and  $\text{P}_2\text{O}_5$  and kept in the powdered form in the dark.

### Water Solubility

The solubility in water was determined at various pH and temperatures by iodometric titration, i.e., saturated solution obtained by shaking the salt for 30 minutes in a thermostat was titrated by 0.001 N thiosulphate (2 ml. of solution with 3 drops of 1 N  $\text{H}_2\text{SO}_4$  and 1 drop of 5 per cent KI solution) in the presence of starch. At 25° the average value of the solubility in water (pH 6.5) was 0.2 millimol. per litre. In 0.01 N  $\text{H}_2\text{SO}_4$  (pH 2.2) the value was 0.116 millimol. per litre but with further increase in acidity the solubility again rose (0.22 millimol. at pH 1.15, 0.5 N  $\text{H}_2\text{SO}_4$ ). The time of shaking (2 to 30 min.) and variation of temperature between 18° and 25° had no appreciable effect on the solubility.

Relation between chloride and iodate concentrations: If  $[\text{IO}_3^-]_e$  is the iodate concentration when equilibrium has been attained,  $[\text{IO}_3^-]_s$  is the iodate concentration due to the solubility of  $\text{Hg}_2(\text{IO}_3)_2$ ,  $[\text{Cl}]_i$  is the original chloride concentration, and  $[\text{Cl}]_f$  is the final concentration, then from the values of the solubility products of mercurous iodate and mercurous chloride,  $[\text{Cl}]_f = 0.0000207 \times [\text{IO}_3^-]_e$ ; also,  $[\text{Cl}]_i = [\text{IO}_3^-]_e + [\text{Cl}]_f - [\text{IO}_3^-]_s$ . Since  $[\text{IO}_3^-]_s$  is

equivalent to  $[\text{Hg}]$  which is determined from the equation,  $[\text{Hg}]^2 \times [\text{IO}_3^-]^2 = S \text{ Hg}_2(\text{IO}_3)_2$ , where S means solubility product, then  $[\text{IO}_3^-]_s = S \text{ Hg}_2(\text{IO}_3)_2 / [\text{IO}_3^-]_e = 0.0256 / [\text{IO}_3^-]_e$ . For an acid medium (pH 2.2),  $[\text{Cl}]_f / [\text{IO}_3^-]_e = 0.000159$ , and  $[\text{IO}_3^-]_s = 3.3 \times 10^{-3} / [\text{IO}_3^-]_e$ .

For various value of  $[\text{IO}_3^-]_e$  from 1 to 12 millimol. per litre, calculations show that  $[\text{Cl}]_f$  is always so small that it may be neglected both in water and in acid medium (pH 2.2), and that  $[\text{IO}_3^-]_s$ , as  $[\text{IO}_3^-]_e$  increases, decreases in water medium from 2.6 to 0.017 per cent of the total iodate and decreases in acid medium from 0.33 to 0.002 per cent of the total iodate. Thus, in acid medium, it is possible to neglect  $[\text{IO}_3^-]_s$  and to assume  $[\text{IO}_3^-]_e$  equals  $[\text{Cl}]_i$  for all concentrations in the range 1 to 12 millimol. per litre. In water the results for the lower concentrations may be 2.6 per cent too high.

### Method and Results

Method of determination: 10 ml. of the chloride solution under test (pH preferably 2.2) are treated in a test-tube with solid mercurous iodate (about five times the stoichiometric quantity) from a spatula; the test tube is closed by means of a rubber stopper, the first portion of the filtrate being rejected. 1 ml. of the clear filtrate is then titrated with 0.01 N thiosulphate.

Results of experiments: In water (pH 6.5) with 2.05 millimol. per litre of chloride (0.12 g. per l.) results of 2.09 millimol. or 0.1224 g. (4 experiments) and 2.10 millimol. or 0.1228 g. (3 experiments) were obtained. Theoretically, the results should be 0.6 per cent too high; actually they were about 2 per cent too high.

In acid medium (pH 2.2) with 2.05, 5.30, and 9.03 millimol. per litre the actual results were 0.6 per cent too low (theoretically, exact results should be obtained). With 1.22 millimol. per litre (0.072 g.) the results were exactly correct (theoretically, they should be 0.2 per cent too high). All these errors are very low; consequently, the determination may be carried out without bringing into consideration the solubility of mercurous iodate.

**World Tin Output.**—Production difficulties in the Far East led the Tin Study Group, meeting recently in Brussels, to revise the world tin output estimate for 1947 to 117,000 tons, as against last October's estimate of 142,000 tons; for 1948, 163,000 tons (198,000); and for 1949, 201,000 tons (218,000). The peak figure of 1940 exceeded 240,000 tons. World consumption is estimated at 140-150,000 tons for 1947; 184,000 for 1948; and 190,000 tons for 1949.

\* From Zavodskaya Laboratoriya, 1946, 12, No. 2, 179-183.

# SEMI-TECHNICAL CHEMICAL PLANT

## Equipment for the Prototype Installation

WHEN a new process has been successfully established on a laboratory scale, involving perhaps some two or three hundred grams of material, it is usually necessary to carry out experiments on a larger scale before proceeding even to "pilot plant" scale. These tests are designed to give, in the first place, larger quantities of material than can be obtained in the glass flask or beaker, and in the second place to give some indication of the difficulties to be expected when the pilot plant is constructed. Clearly, the material from which semi-technical plant is constructed should be the same as that to be employed on a full scale, so that such troubles as corrosion can be tackled at the very outset. Again, the general principles of construction of the plant should be the same as in the prospective large scale plant. Only in such a way can semi-technical plant tests be any guide to the pilot plant and full size plant of the future.

### Prototype Plants

It follows from this that small plant of the type under review may be considered as chemical engineering in miniature, and should be designed with a view to determining such plant variables as heat transfer, resistance to fluid flow, ease of handling in solid or semi-solid form and the like.

As it is probable that plant units will be used for a wide variety of operations, abundant provision should always be made for test points of all descriptions. Thus, a small fractionating column should be provided with sampling and gauge tappings at

every plate, or in the case of packed columns at every few inches of height. Pipe connections should be made with a view to frequent alteration—a golden rule in this con-

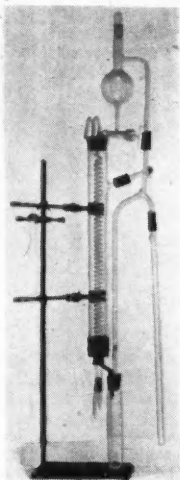


Fig. 2.

nection is "Never use an elbow, but always a plugged tee." Finally, the plant should not be too small.

Although a general rule cannot be made, it is suggested that the smallest convenient working volume will be of the order of five to ten gallons. Smaller quantities involve a number of difficulties due to hold up in pipes, valves and other fittings, while making dimensional similarity in later "scaling-up" still more difficult.

### Structural Considerations

The division of chemical engineering into the so-called "Unit Operations" of fluid flow, distillation, filtration, etc., has been well established for many years, and any description of plant which is based upon the same principles will naturally follow the same classification, especially useful in this case when one piece of plant will be put to a number of uses. Thus, a filter press may be used for a salted out sulphonate in one week, and a precipitated carbonate in the next.

It may be considered good practice to house all the semi-technical plant in a reasonably sized laboratory, one section of which should be carried up to a height sufficient to give a barometric leg discharge. The writer can

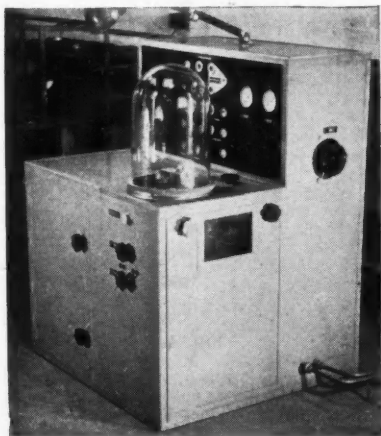


Fig. 1.

speak with some feeling in this matter, having been compelled during the war to open up a skylight and carry a plant up on stilts some ten feet into the air above.

The lofty section of the plant house can be used for distillation columns, which also

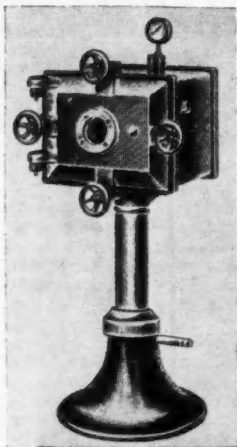


Fig. 3.

make considerable demands for head room. A series of platforms eight feet or so apart, built up from steel girders and removable steel flooring, allow of the building up of any other type of plant which calls for gravitational flow. The flooring at ground level should be of concrete, sloping to a central drain. For building up the framework of temporary plant assemblies, the advantages of bolted perforated girders and beams should not be overlooked.

#### Heat and Flow

Passing on to the actual unit process plant, it is rare to find special equipment built solely for fluid flow or heat transfer measurement. Such determinations of these factors as may be necessary are usually made in the plant built for other operations, making use of the various test points referred to above. This is especially true of fluid flow, where the values required for pressure drop in pipe lines can usually be calculated from the physical properties of the fluid. Nevertheless, where slurries are being pumped, it may be necessary to erect test "pipe-lines" to determine effective viscosity and a unit of this kind, embodying some 60 feet of 0.3 inch bore pipe, has recently been described.<sup>1</sup>

No opportunity should be neglected, while working with the type of plant under review, to obtain the heat transfer rates on which so often the size of the final plant

will depend. While the simpler forms of heat transfer have been exhaustively studied, gaps between theory and experiment frequently occur, especially where non-aqueous liquids are concerned, and experimental figures from small plant are very valuable.

As indicated above, the plant should be designed to admit of accurate temperature measurements at all likely points. If this is borne in mind, there will be no need for specialised heat transfer equipment, except in the case of chemical engineering laboratories, to which reference will be made later. The experimental figures for heat transfer should be determined with due regard to similarity principles, and the heating or cooling media should be the same as that used on a large scale. In pursuance of this principle, electrically-heated vessels are generally undesirable; cooling fluids such as alcohol cooled with solid carbon dioxide should be replaced by chilled brine, and steam heating should be used wherever possible.

#### Small Distillation Plants

A number of small distillation plants are available, embodying a boiling vessel, a fractionating column, condenser and receivers. The lay-out of a unit of this type (shown on a later page) was erected in the laboratories of the Royal Technical College, Glasgow, in the mid 'thirties. This plant has done very good service for the last ten years on a variety of problems, besides being worked in the normal training courses. It is fitted with boiling vessels of two sizes, and designed to work under either atmospheric or reduced pressure. The fractionating column is of the normal bubble cap type, with ten plates, each carrying seven bubble caps. The cross sectional area of the column is 0.2 sq. feet, and distillation rates up to two pound mols. per hour of various mixtures have been

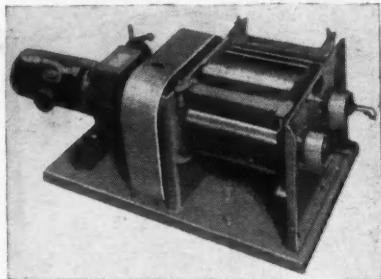


Fig. 4.

obtained. The method of reflux control is by a rocking box, pulled over to supply either reflux or product by one of two electromagnets. These are activated in turn by



a rotating drum, making contact with movable terminals at predetermined intervals.

Were this plant to be re-erected today, the number of bubble caps on each plate would be reduced to one or two, with a cor-

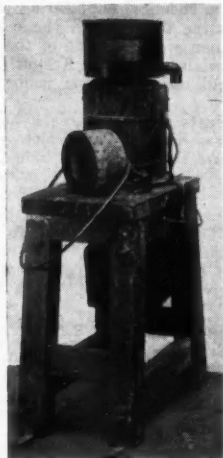
plate, as even with these small columns distinct differences of concentration can be detected across the plate from liquor inlet to outlet, and alternate side downtakes for the overflow are unsound in principle.

The column was constructed in cast iron, clearly the most economic method of fabrication. It has, however, led to considerable difficulty in keeping fluid channels free from rust deposits, this rust being formed in shut-down periods rather than when the plant was working. Where no question of corrosion test is involved, it is possibly desirable to make all semi-technical plant in a more resistant material than iron, in order to avoid this "idle period corrosion."

### 60-70 Per Cent Efficiency

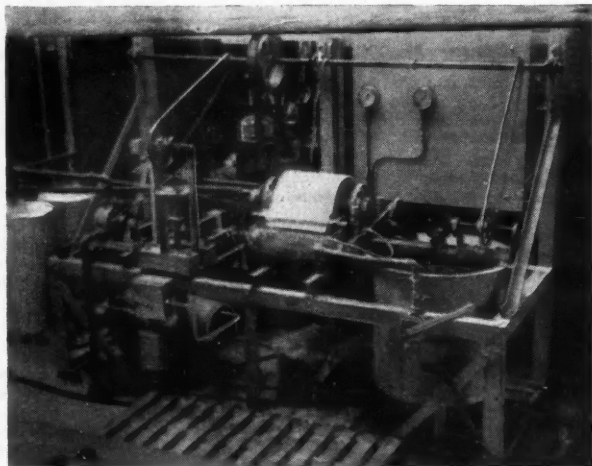
A distillation unit of this type will give plate efficiencies of about 60-70 per cent on normal mixtures, and is clearly inadequate for "close" distillation work unless many more plates are employed. Simpler packed columns, of about the same cross sectional area or less, do not give the poor liquid distribution associated with larger columns of the packed type, and with special packing have been claimed to give at least equal fractionating efficiency to the bubble plate type of similar height and volume.

A very large number of alternative types of packing are available; one of the latest is a modified Raschig ring in wire gauze, with special arrangements to flood the packing before starting a distillation. A small column of this type, twelve feet high and 20 mm. internal diameter, has given an efficiency equal to 450 theoretical plates in the separation of heavy water.<sup>2</sup> It was worked on total reflux, with a boil-up rate of 500 ml.



**Centrifugal Dryer.**

responding increase in size, and the number of plates, and hence the height of the column, would also be increased. Arrangements would be made for the liquid on the plates to flow in the same direction on each



**Fig. 6. The small rotary vacuum filter which may deal with 200 gallons of material per hour and separate up to 200 lbs. of dry solids.**

of water an hour, and mechanically controlled, a matter of great importance in a laboratory where shift workers are seldom available, and where small plant units may take more than 24 hours to reach a working equilibrium.

This type of plant will be working at pressures ranging from one atmosphere to about 5 cms. of mercury pressure. The use of very high vacuum, with pressures of the order of one thousandth of one millimetre of mercury, is spreading, mainly in the purification of complex organic chemicals. The main operating difficulty in these processes is to procure low pressures, and experience from another field is of use here.

### Molecular Still

It is becoming customary, in high grade optical work, to volatilise on to glass surfaces thin layers of sparingly volatile materials, such as silica. This is carried out in a large bell jar-shaped vacuum chamber, exhausted by two-stage oil vapour diffusion pumps, backed by a rotary oil pump. An operating vacuum which shows dead black out to a high tension discharge can be obtained in less than ten minutes, starting from atmospheric pressure. These pumping units can conveniently be attached to any type of "molecular still," as high vacuum units of this kind are usually called.

Fig. 1 shows one of two pumping sets arranged by Messrs. Barr and Stroud for optical work, and the other having been erected for a small molecular still. These molecular stills are simple in design as a rule, and consist of a heating surface over which the oil being distilled flows in a thin film, with a condensing surface at some convenient distance less than the mean free path of the molecule.

Continuous units of the type shown in Fig. 2 have been used for throughputs of the order of two or three hundred millilitres of oil an hour, with the distillant trickling down the spiral on the inner heating tube, and the distillate collecting on the cooled outer tube. It is not known whether the more complicated spinning disc still, devised by Hickman and his co-workers in America, is available in this country, but the construction should not be beyond the powers of any small workshop.

### Gas Technique

Gas absorption and gas washing generally are difficult operations to simulate on a small scale, except in the case of packed tower scrubbers. A number of papers in the American Institution of Chemical Engineers Transactions<sup>3</sup> have described investigations into the operation of small towers of this type, and given abundant details of various types of packing and of flooding velocities for gas and liquid. A choice of packing material can be based on these articles, while

any engineering firm can construct the simple column required from, say, Mannesmann steel tubing with welded-on flanges.

It is possible to obtain flanged glass sections of up to six inches in diameter, and build up a tower for visual study of flow forms, but this would hardly be necessary, except for a chemical engineering laboratory. An alternative to metal in the construction of the column would be one of the synthetic resin materials, such as Keebush, and an entirely acid resistant tower could easily be fabricated.

Where dusts or mists are to be removed from the gas, cyclones and electrostatic precipitators are the most usual industrial practice. The modern tendency is to replace one large cyclone by a number of smaller units in parallel, and one of these small cyclones would serve very well for plant of the type under discussion. They would need to be built to specification, but some useful data are given in an article<sup>4</sup> of a few years ago.

Electrostatic precipitation is quite straightforward, and the author has built successful precipitators with tubes of one inch diameter or less, with a makeshift electrical arrangement. Main difficulties in large-scale practice arise from the accumulation of precipitated material at the electrodes, and it is doubtful whether the small precipitator can give much guidance in predicting large-scale plant performance.

### Reaction Vessels

Simple stirred reaction vessels of many shapes and sizes are readily available, as a steady demand for these comes from many industrial undertakings. In general, unless high rates of heat transfer are required, reaction vessels of the size range from 5 to 50 gallons can be obtained with enamel linings. This allows the use of most acid solutions quite indiscriminately, and the material of the pan has no effect on the chemicals being handled. If high rates of heat transfer are required, the vessels can be fabricated in stainless steel, copper or monel metal.

All these pans are usually fitted with steam and water jackets, and a tipping mechanism is a great advantage. If a number of vessels have to be stirred at intervals, portable electric stirrers are available, very much on the lines of the smaller laboratory types. It should be noted that these stirrers will normally only mix mobile liquids or slurries effectively.

It is not easy to simulate the stirring patterns of larger vessels, and results for times of mixing or heat transfer are not very reliable as a basis for design on a larger scale. The advantage of a number of small reaction vessels, open or closed, in corrosion resistant material, is that they can be assembled in various ways to make up a pilot plant when necessary.



Passing on to evaporation, the evaporator which lends itself most readily to small-scale operation is the Kestner type, with relatively few boiling tubes. The Mirlees model is an arrangement of a compact evaporator of this kind, with one tube up and one down, to give an overall length of 12 feet of one inch diameter tube. The heating surface of some three square feet will give evaporation rates up to 50 lb. per hour with quite low temperature differences between solution and steam. At the same time, the cross section of the tube is quite normal for large evaporator units, and a good indication of full scale working can be obtained.

If more ambitious working, with multi-tube calandria, is required there is no difficulty in obtaining small standard evaporators, of twenty to thirty square feet heating surface, arranged as either single or double effect. Design in this type of plant has gradually been changing towards external calandria, with liquor-vapour separators instead of the large evaporator bodies, and forced circulation is quite common. For a small unit of the type under consideration, this design is particularly suited.

#### Drying Plants

Where drying plant is concerned, the simplest full-scale unit to duplicate on a smaller scale is the vacuum shelf dryer. A small dryer of this type is shown in Fig. 3 and has two steam-heated shelves, each 18 in. by 12 in. in area. The moisture passes to a vertical condenser and receiver built into the base, but a subsidiary vacuum pump is required. Evaporation rates up to 5 lb. of water per hour can be obtained, while the

trays used to hold the material to be dried can be made of enamelled iron or wire reinforced glass.

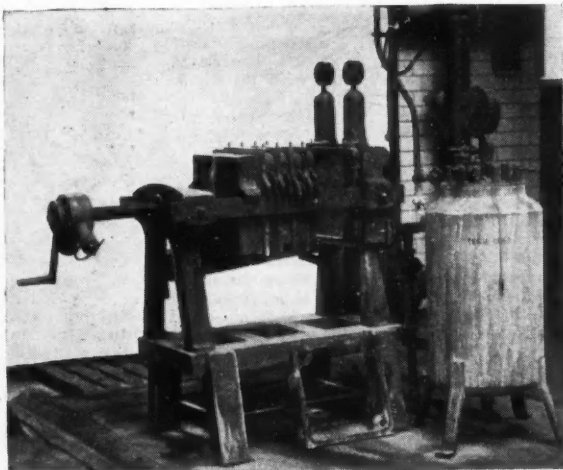
The results obtained by drying in such a unit can be applied directly to large-scale design. Of course, the time and labour saving rotary types of dryer are preferred wherever possible in large-scale practice, but as far as is known, no small rotary dryers have been installed in semi-technical laboratories.

On the other hand, drum dryers, drying a film of material spread on the surface of a steam-heated drum, have been made up in sizes as small as 6 in. in diameter by 6 in. long. An open drum dryer of this type is shown in Fig. 4 but it is quite simple to build a vacuum casing round the drums and operate at reduced pressure. In these small units, it is usual to have two rolls fed from the top centre, and to make the actual surface in stainless steel. The throughput of solid on such a dryer can be as low as 2.3 lb. an hour, but the operation must be carefully watched and controlled to give an even film.

Spray drying necessitates the use of a fairly large drying chamber into which the sprayed material can be blown, but these are now constructed very simply in sheet metal, and one has been made as small as 3 ft. in diameter by 4 ft. deep. Such a unit will deal with a concentrated solution or slurry at a rate of about 20 lb. per hour, giving results which are reasonably representative of full-scale practice.

Centrifugal dryers may be briefly mentioned here, although, removing moisture by mechanical force, they can more logically be classed with filtration units. It is a definite

Fig. 7. Plate and frame or recessed plate filters, such as this, reproduce fairly exactly in miniature the practice employed in larger industrial installations.



advantage to make centrifugal units of small diameter if high centrifugal effects are required, and the various Sharples units referred to later are illustrations of this principle.

A small centrifugal dryer of six inches or so in diameter presents no difficulties. This unit is of conventional design, with a bowl 6 in. in diameter, revolving at 6000 r.p.m., and capable of dewatering about a pound of sandy crystals down to 1-2 per cent of moisture in five minutes or less.

#### Four Types of Filter

There are four important types of filter unit in large-scale practice—the rotary vacuum filter, the plate and frame filter press, the pressure leaf filter, and the edge filter. All these have been duplicated on a pilot plant scale, and are capable of giving results which can be applied to larger scale design.

A small rotary filter assembly is shown in Fig. 6. The actual rotor is 1 ft. in diameter by 1 ft. long, and will give overall rates up to 100 gallons per hour with free filtering slurries. The filter cakes can be built up with ease to  $\frac{1}{2}$  in. in thickness, and outputs of the order of 200 lb. per hour of dry solid may be obtained. A Dorr type sludge pump circulates the slurry from the stirred tank on the right of the assembly through the trough of the filter and back to the tank at a rate of 200 gallons per hour, while a system of coned pulleys allows a wide variation in drum speeds. A vacuum pump with a capacity of 12 cu. ft. of free air per minute maintains a vacuum of 15 in. of mercury for typical filter cakes.

Actual operation is very smooth but the filter cloth tends to choke more rapidly than in large-scale practice. It is probable that a string discharge, which can be fitted to these filters without difficulty, would improve this working detail. It is interesting to note that the performance of various slurries in this filter can be predicted with reasonable accuracy from tests on a small suction leaf filter of 4 in. diameter, which is lowered into the slurry to be tested.

#### Most Practical Size

Plate and frame, or recessed plate filters, follow the design of industrial filter presses faithfully, as will be seen from Fig. 7, showing a press with 12 in. square plates and frames. It will be noted that a monteju, or pressure egg, is used for forcing the slurry into the press. On a small scale, with only one or two frames filled, a monteju will hold sufficient slurry (about 15 gallons) at one filling without being inconveniently large. Small pumps always give maintenance trouble, especially when used intermittently as in this case.

Smaller presses than the one shown are available, the author having used one in hard lead, with plates 3 in. square, while a 6 in.

square type is recommended by one maker for testing purposes. These very small presses, however, have the relative size of ports, liquid channels and filter surfaces rather distorted, and it is suggested that a 12 in. square unit is the most desirable size.

Just as in larger plant, these filters can be constructed from any mechanically sound material, such as bronze, stainless steel, or hard wood. The one shown is in acid-resisting bronze, with frames of thickness from a half to one inch; composite frames up to 2 in. in thickness have been built up, and it would be easy to construct a 4 in. thick frame if desired. The press is equipped for steam heating and for through washing, as in normal industrial practice.

The normal form of plate and frame filter is designed to remove large quantities of solid from a slurry; when the solid represents a very small fraction of the slurry, the operation is one of clarification, usually from very finely divided solids. This can either be by edge filtration, or by a special form of recessed plate filter press, carrying filter plates of asbestos or some similar material. This is designed to remove bacteria, among other matters, and the filter is generally made small enough to be put bodily into a sterilising oven. A 9 in. square filter of this type, with four asbestos plates, was much used in connection with blood transfusion work during the war.

#### Two Other Types

Edge filters, or filters made up from a number of filter discs pressed together, with a central channel into which the clear filtrate is forced from the outside, are never made up with very large discs, a customary industrial size being about 2 in. in diameter.

For industrial use, a number of these packs are mounted on a common header inside a large pressure tank. Clearly the smaller unit, containing two or three filter packs in a smaller pressure tank, is a very close simulation, and such filters are available. In fact, owing to their extensive use in the reclamation of small amounts of used lubricating oil, these small edge filters are more often constructed than the larger industrial units.

The fourth type of filter, the pressure leaf, also demands a pressure tank, and both this and the edge filter are best served by compressed air and a monteju, rather than pumps.

A small pressure leaf filter, with a total filtering area of 0.5 sq. ft. is shown being discharged in Fig. 8. It will be seen that the cake has grown round the flat leaf and is gradually assuming an hemispherical form. It might be better to choose a slightly larger unit, so that the ratio of filtering "edge" to filtering "side" was more like that obtaining in full scale units. At the same time, the size shown is very convenient

for small scale working, and the advantage of easy cleaning which this type of filter possesses should not be overlooked.

### Crushing Equipment

Grinding plant for semi-technical work will not have to deal with materials of more than 2 in. in size, for larger fragments would mean more material than is contemplated here. Ordinary jaw crushers, reducing a 1-2 in. feed to a  $\frac{1}{2}$  in. product, have long been available, while a small roller mill, with two rolls of 6 in. diameter, can reduce such a substance to about 10 mesh or a little smaller.

The only operating difficulty here is the noise and vibration caused by the plant, which may be a source of considerable annoyance. In these circumstances, it is better to house the crushing plant in a separate room, or, if possible, in a small hut detached from the main building.

Further size reduction is best carried out in a ball mill or high speed disintegrator. As far as is known, the very important medium speed roller mills of the Sturtevant or Raymond type have not been built on the small scale being considered.

Ball mills have long been available in small sizes; as an example, the author has used porcelain ball mills of about one pint capacity, working with porcelain balls, to grind soft crystalline material for laboratory testing, and has seen an exactly similar arrangement with a pot mill of 10 gallons capacity.

Steel pots working with cast iron or steel balls, can be just as easily obtained, and special mountings for a number of these

driven at one time are available. As a rule, these mills are for batch working, but a hollow trunnion at each end can allow them to be used in continuous grinding. A typical mill of this kind has been used for both wet and dry grinding in continuous flow, although it is only 6 in. in diameter by 20 in. long.

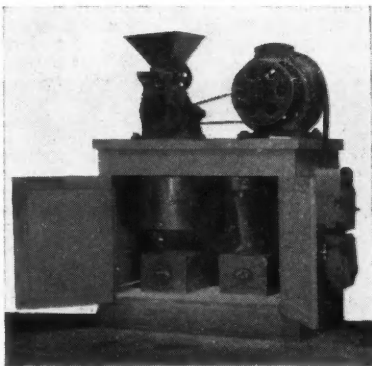


Fig. 9.

With wet grinding, a throughput of 1-2 lb. per hour reduced from 10 mesh to below 60 mesh can easily be obtained. Dry grinding of cannel coal, with an air-swept mill, gave 200 grms. per hour of sub-120 mesh from a 10-mesh feed.

High speed mills can be either of the swing hammer or the squirrel cage type, with rotors of about 6 in. in diameter, and have the advantage that they are self cleaning by induced air draught. This, combined with a very small working volume, means that they can be used conveniently for relatively small quantities of material, though the actual throughput per hour can be high.

By using these mills with a screen and proper means for collecting the fine dust, a very finely divided product (all through 120 mesh in one typical case) can be obtained. A Pulmac mill, mounted above a cyclone precipitator and dust filters is shown in Fig. 9, and has been found to work very smoothly, although a subsidiary fan to draw air through the mill, over and above the self-induced air, is an advantage. This 6 in. mill can give up to 50 lb. per hour of under 50 mesh material from a fairly soft rock of  $\frac{1}{4}$  in. size.

### Making Emulsions

While these grinding units will reduce material down to about 5 mu, it may be necessary to disperse this powder very thoroughly in liquids, or to create a liquid dispersion of the same particle size. This requires a high-speed rotary colloid mill or a high pressure homogeniser; both of which



Fig. 8.

can be made up in small sizes. Thus a Premier colloid mill can be obtained with a rotor as small as 3.5 in. in diameter, and a throughput of 15-20 gallons per hour. It will give commercial emulsions and pastes from a wide range of components.

The alternative form of homogeniser is usually made up as a reciprocating pump, delivering against a pressure of the order of 2000 lb./sq. in. A unit of this type made by G. and J. Weir, with three  $\frac{3}{4}$  in. pistons in parallel, can give an emulsion from oil and water at a rate of 100 gallons per hour, with no oil droplets above 5  $\mu$  in size.

A testing sieve shaker, besides acting as a check on larger scale working, can be used as a semitechnical sieving unit. A shaking system with a series of 6 in. diameter sieves will deal with about 20 bl. of material graded from 5 mesh down to 100 mesh in an hour. Small classifiers of the Dorr type would need to be specially made, and although the author has used a small water classifier of the Andrews spiral type with a 9 in. diameter cone, this had been made up for trial only and not as a standard appliance.

If the material to be classified is very fine—below 100 mesh—then the high-speed centrifugal will enable a series of samples of varying size to be collected by altering the time of flow through the unit. A Sharples centrifuge of laboratory size will probably be large enough for most of the requirements of the semi-technical laboratory. It can also break down the troublesome emulsions which are easily formed when such processes as the washing of hydrocarbon oils are undertaken.

All the unit operations of technical chemistry can be attempted satisfactorily with the plant described above, and a range of these units would satisfactorily introduce the student chemical engineer to the idea of plant control and operation.

If it is desired to build up a laboratory for this purpose, then even more test points than have been suggested should be introduced at all likely section of any plant. All such units as steam traps should be provided with a two-way outlet, one to drain and the other to a weighing stand.

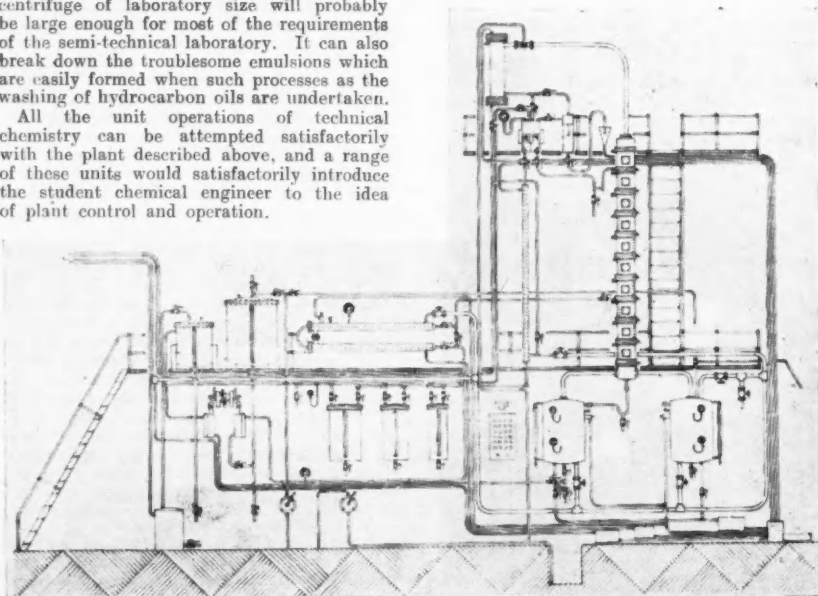
A balance of the steel yard type, weighing up to 5 cwt., with accuracy to the nearest pound, and one or two scales of smaller size will be required, with a number of flow gauges of various types. Controls of complicated pieces of plant should be brought to a central point, as in commercial practice, and if possible some arrangement for automatic control should be introduced. At the same time, as many alternative methods of operation as possible should be provided; as an instance of this may be cited a refrigeration unit in which the refrigerant can be expanded either through an automatic, thermostatically controlled valve, or through a hand-operated manual control. This has been installed recently in a college laboratory by Messrs. L. Sterne of Glasgow.

<sup>1</sup> *Trans. Amer. Inst. Chem. Eng.*, 1946.

<sup>2</sup> *Nature*, 1946, 158, 164.

<sup>3</sup> e.g. *Trans. Amer. Inst. Chem. Eng.*, 1945, 41, 698.

<sup>4</sup> *E. M. Larcombe, Ind. Chem.*, 1942, 15, 433.



Layout of the small distillation plant, referred to earlier in this article, which has rendered good results at the Glasgow Royal Technical College.

## New Use for Hydraulic Control

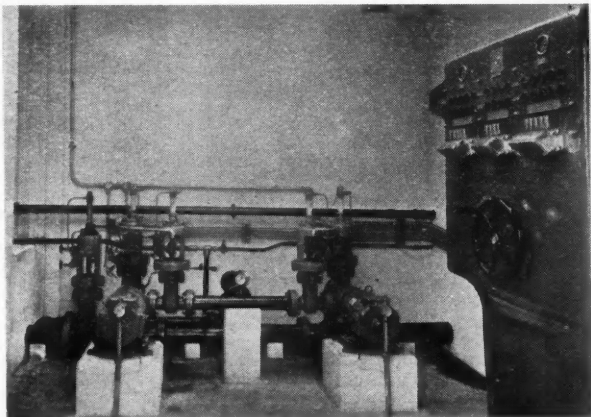
### Innovation at Southampton Waterworks

**A**LONDON engineering firm, the Paterson Engineering Co., Ltd., 31 Windsor House, Kingsway, W.C.2, has applied the well-known "Lockheed" hydraulic control, so extensively used in connection with motor car brakes and aircraft controls, to

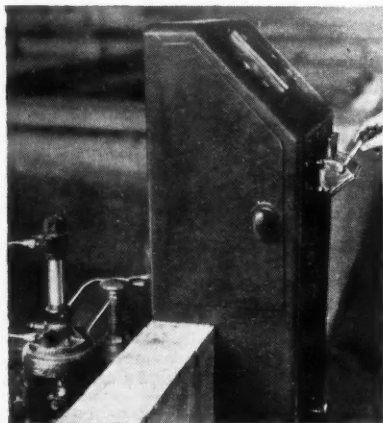
at Otterbourne, dealing with 3,500,000 gallons per day of river water, this being the first section of a 10,000,000 gallon per 24 hour plant.

The "Lockheed" system is here shown to be particularly applicable to rapid filter

Lockheed operated valves on sedimentation tanks and sludge pumps, showing, on right, the control panel.



the hydraulic control of water purification plant. An excellent example of their methods is at the filtration plant of the Southampton waterworks pumping station



The new method of control of filter valves is operated from this cabinet.

valves which are specially prepared for the mounting of the actuating cylinder, the piston rod being connected by a fork coupling to the valve spindle. The valves on each filter are controlled from a central cabinet placed so that as the valves are operated the result can be seen, particularly during the cleansing process.

When one motor-operated pump is used, push buttons are provided on each cabinet for stopping and starting the pump. The latter is capable of delivering fluid at pressures of about 700 lb. per sq. in., and even at this high pressure the equipment, in case of current failure, is easily operated by hand at the required speed of about 120 strokes per minute.

The advantages of the system are (1) ease of installation, (2) small size of the pipes on account of the high pressures used, (3) minimum maintenance as the hydraulic fluid is self-lubricating, (4) the fluid will not freeze at temperatures over minus 40°C., the whole system is self-contained with its own pressure pump and supply tank, (5) the cylinders are of the double-acting type fitted with a self-locking device so that as soon as the operating pressure is removed the piston is locked in place and can only be moved again by application of further pressure.

## Progress in Instrumentation

### Current Technique in German Chemical Plants

THE German chemical industry made progress in the war years in many directions and not least in the science of instrumentation. Several of the new forms of German instrumentation deserve very careful study by the industry in this country, especially, perhaps, their magnetic oxygen recorder employed to determine oxygen in gas mixtures.

This is one of the subjects dealt with in "Instrumentation and Control in the German Chemical Industry" (BIOS Final Report No. 1007) which is divided into several sections covering such subjects as flow, pressure, temperature, density, analysis measurements and automatic control. The only general criticism which has to be made concerns the inadequate treatment of spectrographic methods, a technique well known in German industry. It is possible, however, that this system of control was not used to any great extent.

#### Oxygen Determination

The determination of oxygen in gas mixtures has so far normally been carried out by one of the following three methods.

(1) Determination by means of the heat of reaction. This is the most useful method for evaluating small quantities of oxygen, but it suffers from two disadvantages. The first is that the catalyst may be poisoned by impurities such as sulphur, and the second is that the catalyst may be sooted up by unsaturated hydrocarbons. (2) Determination by combustion or chemical absorption of the oxygen. (3) Determination by measurement of changes in conductivity resulting from chemical reaction of the oxygen.

The determination of oxygen by means of a magnetic recorder is based on the fact that oxygen has a higher magnetic susceptibility than other gases, and it is paramagnetic while other gases are diamagnetic. Nitric oxide is, however, an exception to this general rule. The method was used in Germany for the determination of oxygen in acetylene and butadiene.

Briefly, the principle is as follows. The gas, of which the oxygen content is to be determined, is heated in a strong non-homogeneous field. As the magnetic susceptibility of the gas alters with rise in temperature, the attractive force exerted by the magnetic field on the gas will also change. The result is that a flow of gas is set up, the direction and strength of which gives a measure of the oxygen content. The gas flow is measured by temperature-sensitive elements which are heated or cooled by the passage of the gas over them.

The details given in the report are

sufficient to enable anyone interested to construct an apparatus suitable for this purpose, but it appears fairly clear that considerable development work is required before such an instrument could be relied upon to give accurate results. The theory of the apparatus and various examples of its use are adequately described in the text.

A further interesting process is that in which carbon dioxide is measured in a mixture of gases in which hydrogen is also present. The gaseous mixture is passed through a heat-conductivity apparatus in which there are four gas cells. Each cell contains a platinum wire and the four cells are connected to the four corners of a wheatstone bridge. The gas under observation is passed through one pair of cells connected to the opposite corners of the bridge, while a comparison gas passes through the other two cells.

By taking measurements before and after absorption of carbon dioxide from the working gas and comparing them with measurements obtained from the comparison gas, an estimate of the carbon dioxide content of the working gas may be obtained. Provided there is at least 25 per cent of hydrogen present, the carbon dioxide measurement is independent of hydrogen concentration.

#### Infra-Red Meters

For temperature measurements up to 500°C., German technicians appear to prefer resistance thermometers to the thermocouple. Infra-red absorption meters are in use in several German chemical factories, the advantage of this type of instrument being that complicated gas mixtures can be evaluated satisfactorily under conditions where this could not be done by other methods.

The infra-red absorption meter functions on the principle of measuring the degree of absorption effected by a sample of gas on infra-red radiation passing through it. The actual analysis of a complex mixture of gases is extremely involved and it is not possible to discuss, in a short review of this type, all the details outlined in the BIOS report.

Other items dealt with include pressure recorders for gases, liquid level gauges, apparatus for recording density measurements of liquids and gases, and automatic mechanisms for controlling weighing operations. The investigators are to be congratulated on producing a report that will be of use to many branches of British industry—subject to the customary proviso that the subject matter may be protected by British and/or U.S. patents or patent applications.



## Electrical Measurements

### Wireless Firm's Laboratory Aids

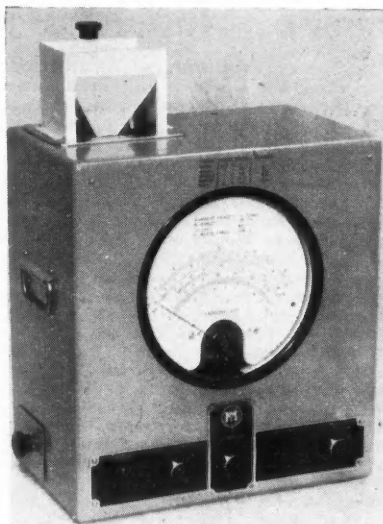
OF special chemical interest among the wide production range of the Mullard Wireless Service Co., Ltd., Century House, Shaftesbury Avenue, London, W.C.2, is a robust, self-contained potentiometric titration apparatus. This, known as the Type E920, is sufficiently simple to be used in routine testing by unskilled operators, and is yet capable of meeting the requirement of the industrial research chemist. It is operated from the 50-c/s. supply mains and the end-point is detected by a "magic-eye" indicator. Thus there are no batteries to be replaced and no delicate galvanometer to be damaged by mechanical shocks or electrical overloads. A special circuit eliminates all possibility of drift during a titration and changes of mains supply voltage do not give rise to any inaccuracies.

The potentials from the electrodes in the solution are applied to the terminals on the potentiometer unit, and the potentiometer is adjusted until the area of fluorescence on the screen of the "magic eye" is a minimum. The value of the applied potential difference is then read off directly from the calibrated scale. A sensitivity control is provided, and at maximum sensitivity a p.d. of 2 mV can be detected. The main dial covers the range 0-400 mV, while a five-position switch enables the range to be extended to a maximum of 2 V. A switch controls a subsidiary circuit which provides a polarising voltage for one electrode of a mono-metallic system. A multi-way socket at the rear of the instrument provides power to operate the titration unit.

### Moisture Meter

The need for accurate measurement of moisture content is well served by another Mullard production, the Type E910 moisture meter, which, developed chiefly in the interests of millers and farmers, can be used for any granular substance. This instrument makes use of the capacity change produced between two plates of an electrical condenser by the insertion of a weighed quantity of material. As the dielectric constant is dependent on the moisture content of the substance, the measurement of the capacity with the sample *in situ* is an indication of how much moisture is present.

The instrument is calibrated directly in percentage moisture content, four scales being provided for cereal samples, and an arbitrary scale graduated 0-10 when it is desired to measure the moisture content of any granular substance other than grain. Provision is made whereby the setting of each scale may be checked against an internal standard, and, in conjunction with



The Mullard moisture meter which determines by electrical means the water content of any granular substance.

a special filling device and the supplied temperature correction chart, measurements can be repeated with an accuracy of  $\pm 0.2$  per cent moisture. The apparatus is entirely self-contained and operates directly from a 50-c/s. mains supply.

### Measuring Metallic Grains

A NEW contribution to the search for better metals and by-products of metallurgical studies is the new grain size comparator developed by F. C. Hull, of the Westinghouse Research Laboratories, Pittsburgh. Consisting of a ground-glass screen hinged to an illuminating unit with a slotted wooden frame, it is easily attached to a standard metallograph.

To determine the grain size of a particular specimen, a polished and etched sample is placed on the stage of the metallograph and projected in magnified form on the glass screen. Its granular boundaries can be seen in clear, map-like form. A transparent slide of a known standard grain structure is slipped into the wooden frame and illuminated by incandescent light. The magnifying power of the metallograph is then changed by extending the bellows until the unknown image matches the grain size of the standard. The amount of adjustment needed is read from a scale on the metallograph, and grain size of the unknown specimen determined by reference to a graph.

## Australia's Science Review

### Melbourne Exhibition Reveals Great Progress

**H**OW rapidly scientific technology has advanced in Australia, particularly during and since the war, is not widely realised, even in the southern Continent itself. The elaborate exhibition of chemical and instrument technology—"Chemex—1947"—which the Australian Chemical Institute and the Society of Instrument Technology are currently holding in Melbourne's Exhibition Building is well designed to awaken interest in Australia's scientific progress and to illustrate how far it has progressed. Although this is not purely an Australian exhibition, for it includes a general survey of the state of industrial science in the world, many of Australia's special achievements in chemistry and instrument technology are illustrated in the exhibits by private firms, manufacturers and importers and Australian public institutions.

#### Popular Exhibits

The exhibits which are gaining the most lively public interest are those which, for the first time in Australia, reveal in model and diagram some of the chief wartime secrets. The Royal Australian Air Force has a show of radar equipment, armament, air sea rescue equipment, and—biggest draws of all—a V-1 "Buzz" bomb and a "Derwent" jet engine, a gas jet turbine made by Rolls Royce of the same type as those which drove the record-breaking Gloucester Meteor. The biggest attraction for those with inquiring minds is the atomic energy exhibit set up on the stage. Although this exhibit necessarily stops far short of showing all there is to show about atomic energy research, it traces by means of models, graphs, charts and diagrams the early experiments in atom fission that led to the making of the atom bomb, and sketches in some of the possible peacetime uses of atomic energy. This exhibit has been built up under the supervision of Professor L. H. Martin, who has charge of Melbourne University's fine new atom research laboratories.

#### Science and Industry

The Council for Scientific and Industrial Research shows the work of two of the dozens of laboratories it maintains throughout the continent for investigating problems in primary and secondary industry. The Council's Division of Industrial Chemistry shows the magnetic separation of minerals from the black sands of the north-east coast. These sands form one of the world's greatest mineral deposits; they contain monazite, a source of thorium and rare earth metals; rutile, which gives titanium; ilmen-

ite, a compound of iron and titanium; and zircon, from which the metal zirconium may be obtained.

The Industrial Chemical Division also shows the flotation process of separating valuable minerals from the ores. Australian mining companies were the first in the world to use flotation on a large scale, and the Physical Chemistry Section of the Industrial Chemistry Division is constantly at work studying the process and applying its findings to the development of Australia's mineral resources. During the war, most of this section's attention was given to the concentration of strategic minerals; cassiterite, scheelite, fluorite, zircon, and rutile.

The Tribophysics Section of the C.S.I.R. has made a hit with its Photocell chronometer, an electronic instrument that measures the muzzle velocity of projectiles with a maximum error of 0.2 per cent. The Tribophysics Section developed this instrument during the war, and it has been used by both Army and Navy. The principle employed is not new, but use of an electronic crystal controlled counter allowed the distance over which the projectile is timed to be reduced to ten feet, and so for the first time a portable instrument was produced. The lenses and other optical parts were designed and made in an optical munitions annex at Hobart, Tasmania.

#### Versatile Plastics

It is evident that plastics have come to stay. Two firms of plastic manufacturers—one of them describes itself as the largest in the southern hemisphere—have stands at the exhibition and are showing an extraordinarily wide array of plastic materials and articles, from kitchen ware, garden hose and boot uppers to switchboard panels and motor-car parts. One of the firms makes a synthetic resin cement used as the main adhesive in building the famous Mosquito fighter-bomber. Very nearly all the raw materials used in this industry are of Australian origin.

Several Australian drug houses demonstrate in their exhibits the many innovations in drug manufacture mothered by an isolated Australia's wartime needs. One firm shows a model alkaloid extraction plant, of the kind developed to extract atropine and hyoscyne from the leaves of an Australian tree, the *Duboisia*, whose peculiar properties were known to the Australian black men long before necessity drew the white man's attention to it. Hyoscyne was most valuable in war for the treatment of shock and hysteria, and Australia was sole supplier to the United Nations.



## American Chemical Notebook

*From Our New York Correspondent*

**M**ONSANTO Chemical Co., St. Louis, Mo., at the annual meeting announced plans to produce new post-war products at an estimated cost of \$50,000,000. The company's previously announced \$22,000,000 construction programme is now under way. Declaring that only a "small amount" of the new construction represents additional plants for old-line products, the chairman added that these developments "do not seem to be the end." While complete details of the vast construction programme were not revealed, the company disclosed that Monsanto's research department had developed fifty-two new chemicals last year, several of which will be manufactured in volume under the new plans. Among the projects is the construction of a \$10,000,000 styrene plant for volume production of polystyrene plastics to be erected by Monsanto in Texas City, Texas. At Everett, Mass., plants will be erected for the production of new textile chemicals, including resloom, which prevents woollens from shrinking, wrinkling, or losing shape, and syton, which strengthens cotton, and reduces slippage of rayon and nylon. Also included in Monsanto's plans is the completion of the world's first manufacturing plant for the production of synthetic caffeine. At Trenton, Mich., the company will construct new facilities for producing Monsanto's synthetic detergent, sterox or sudsless soap. At the meeting, the president told stockholders that Monsanto's sales attained nearly \$100,000,000 in 1946, and that profits, after taxes, were twice as large as in 1945. He also reported that in the last ten years the company's sales have increased 250 per cent. With regard to the future, he said that "in spite of the nation-wide general price increase, we expect the average level of our 1947 prices not to exceed 85 per cent of the level in 1926."

According to a report prepared by S. G. Newman, of the U.S. Army Ordnance Department, the best alkaline immersion cleaner for rust-resistant phosphate coated metals is sodium metasilicate pentahydrate. Tests at the Springfield (Mass.) Armoury have revealed that a concentration of 6 to 10 oz. per gallon of water will not harm protective phosphate finishes and at a concentration of 6 oz. per gallon the compound does not corrode light metals or zinc die castings. The degreasing effect of alkali immersion cleaners is greatly improved by alkyl aryl sodium sulphionate, a surface active agent, the report states. Addition of 10 per cent of the sulphionate to the pentahydrate solution gives best results. The report recom-

mends immersion of greasy metals in such a solution for about 10 minutes at 205° to 210°F.

\* \* \*

The French Supply Council informed the Civil Production Administration that shipments of French potash would be resumed this spring. Imports of French potash into the States has been cut off since 1941 and the present move is expected to boost the short domestic supply during the fertiliser period. The schedule sent by the French agency to the Civil Production Administration calls for shipment of 5500 short tons of potash and basic potassium oxide in March; 5500 in April; and 5000 in May. C.P.A. asked the French Government to expedite deliveries against its international commitment, made through the International Emergency Food Council, when it became apparent that the States' domestic supply of agricultural potash for April-May would be 25 per cent below what was available in the same months last year. Before the war enough potash was customarily shipped from France and Germany to the States to meet approximately 50 per cent of domestic requirements for fertiliser. Though domestic potash production has more than doubled since imports from Europe were cut off, it has not reached the level to which demand has "rocketed" since the beginning of the war. Sales of the imported French potash will be made through the French Potash and Import Co., Inc., New York, and will not be subject to domestic allocations.

\* \* \*

Babcock & Wilcox Co., New York, announces a programme of expanded research, which will be centred in a building on a 20-acre site near Alliance, Ohio. The programme will include generation of steam, combustion methods, utilisation of fuels, metallurgy and welding, refractories, and other fields closely connected with the company's extensive manufacturing programme.

**Colombian Imports.**—By a decree dated March 13, it is laid down that if goods enter Colombia by a port other than that specified on the import licence, the importer is liable to a fine of from ten to one thousand pesos.

**U.S. Steel.**—Production of steel by domestic mills during March established a peace-time record of over 6,500,000 metric tons, bringing the first quarter's output close to 20,000,000 metric tons, also, the highest for any peace-time quarter, the American Iron and Steel Institute has reported.

## NOTES FROM HOLLAND

*From Our Own Correspondent*

*Amsterdam*

**A**CENTRALISED sale agency for chemicals is now being set up by a number of well-known manufacturing concerns here under the style of "Verkoopkantoor voor Chemische Producten." This agency will deal with such products of the Limburg States Mines, the Sulphuric Acid Manufacturers Ketjen, Ltd., the Royal Dutch Salt Industry, Ltd., and the Mekog Co. for Gas Exploitation, Ltd., as are not covered by other organisations. The concerted selling action does not prejudice the independence of each partner, but aims at increases of efficiency and rationalisation in marketing of products.

The monthly output of crude petroleum at Schoonebeek by the Royal Dutch-Shell concern has been increased to about 15,000 tons. According to official statements, a further rise to 25,000 tons may be expected. But experts seem to doubt further big progress.

The Hague Ministry of Economics estimates the Dutch salt output in the current year at 230,000 tons, as compared with 200,000 tons in 1939. Salt mining will be concentrated at Hengelo, so that production at Boekolo will be abandoned in the near future.

### Chemical Exports and Imports

While the ratio of Dutch exports and imports of chemicals has thus far remained unchanged in value in the last year—i.e., 50:100—compared with pre-war years, there have been marked shiftings in some single items. The export of saccharin was before the war of little importance, but it increased more than thirty-fold in 1946 in weight (and, of course, much more in value). Ammonium chloride, dyed lacquers and varnishes, caraway oil are among the active individual items. There are practically no imports of blanc lacquers and varnishes and Holland has also become more than self-sufficient in ammonium chloride. The need for the ironfree quality is being covered by the refining of crude sal-ammoniac. There is at present a surplus production of printer's ink, which is earmarked for export. Roughly speaking, the persistent "bottlenecks" of coal and raw material scarcities are still checking the former active lines—e.g., paints and dyes, linoleum, glue, essential oils and perfumes—from coming again into their own. Nevertheless, it may safely be said that the Dutch dye industry did not suffer too much from war devastation. Sixteen dry paint factories, of which five white lead, two white zinc and one lithopone, and eight mixed factories turned out in 1946 some 29,000 tons

at a value of more than 5 million florin and exported some 3500 tons (to Sweden, Belgium, Switzerland, Finland) at a value of 4 million florin, while the total costs of the imported raw materials were about 2 million florins. It may be noted in passing that the *Export Groep Verf* (Export Union for Dyes), formed towards the end of 1945, refers only to dressed dyes, not to pigments and dry paints.

### Distribution of Tar Products

The Hague authorities aim at freeing at an early date the distribution of tar products, which are still largely—with certain notable exceptions—subjected to the controlling competence of the *Rijksbureau voor Teepproducten* (National Bureau for Tar Products), which disposes of the products from tar turned out by the blast furnaces of IJmuiden, the coke batteries of the State Mines of Limburg province, as well as by some 160 town gas plants. Under the "National Tar Contract," all tar producers are bound to deliver the tar exclusively to the combine's tar distilleries. In the present phase of Dutch rehabilitation there is, of course, a heavy scramble for mastic, tar paper and way tar; but pitch, tar oils, naphthalene, anthracene and benzol products are at least of the same immediate importance. So the task of the Government in directing the flow of tar distillation for special purposes is by no means easy, especially as the coke installation of Sluiskil was destroyed in the war, while the Dutch production of crude tar amounts now only to some 50 per cent of the pre-war period. As a matter of fact, reconstruction plans have to be curtailed by lack of building materials from tar, nevertheless regard is taken to ensure the regular production of, say, phenol or cresol for the young plastic industry, or of a number of other fundamental tar chemicals which are now playing their rôle in the conclusion of commercial treaties between Holland and other countries.

The conference on "Research and the Smaller Firm," held in Manchester last October under the auspices of Manchester Joint Research Council, is now summarised in booklet form, including texts of the papers prepared for the conference and written contributions since received. Published by and available (2s. 6d., plus postage) from the Council at Chamber of Commerce, Ship Canal House, King Street, Manchester 2, it affords interesting commentary on an important topic.

# BEET SUGAR MANUFACTURE

## New Italian Process

**I**N Dr. Bonelli's new patented method for extracting sugar from beet the roots are cleaned (defecated), reduced to a pulp with lime not exceeding 0.8 per cent and the pulp then subjected to a first pressing, repeated washing, and a second pressing. The treatment with lime in the cold is said to ensure greater purity of the juice than in the usual diffusion process. The juice is then heated, further refined with 0.1-0.3 per cent lime, saturated with carbon dioxide, filtered, and finally boiled and centrifuged in the ordinary way. Several advantages are claimed in comparison with the usual diffusion method: elimination of diffusion unit with its corresponding consumption of steam; considerable reduction in amount of water required; less waste of beet and better yield of sugar; and, what is of special importance, reduced consumption of fuel and labour. It is also claimed that the residue has a higher content of dry matter and therefore of nutrient, and that, generally, the whole process is simpler and more efficient.

### Industrial Tests

Although tests on a large industrial scale have not yet apparently been made, those of a semi-commercial or pilot plant nature have been undertaken at the laboratory of the Consorzio Nazionale Produttori Zuccheri, at Pontelagoscuro, and results reported by Mario Borghi (*La Chim. e l'Ind.*, 1946, 28, 177-184).

Subject to further large-scale test Borghi is of opinion that the new method may be a fundamental advance in beet sugar manufacture. He thinks the greater purity of the juice is due to the action of lime both on the cellular tissue and on the juice, so that the former is more easily removable. In the methods commonly used to-day the pectic matter, during hot diffusion, is only partly precipitated by hot defecation or refining. Much of it passes through all stages into the final molasses. With lime treatment, in the cold, on the other hand, this material is almost wholly precipitated; and the other impurities in the juice—including albuminous and nitrogenous matter—remain practically unchanged, only reacting to a slight extent with lime in the cold. In the successive phases of saturation and defecation in the cold they follow their normal cycle of coagulation and precipitation.

Admittedly, the greater purity is an advantage, but the final behaviour of organic matter, especially the nitrogenous, requires further experimental study. It seems that the molasses finally obtained may contain fairly high percentages of lime salts, but

it is not thought that this would be a serious inconvenience. The tests at Pontelagoscuro are described at length and results tabulated, with particular attention to the respective merits of single and double saturation with carbon dioxide.

### Results of Experiments

The first tests were made in 1943. Two years later, in 1945, Dr. Bonelli carried out some further experiments at the Zuccherificio di Casalmaggiore, in collaboration with Dr. C. Grossi of the Saccarifera Lombarda. Pre-defecation was done with 30 per cent water containing the desired proportion of lime, followed by a single wash with 20 per cent water. A second defecation of the juice followed, and then either single or double saturation. The amount of lime in this case was increased to see if better purification could be obtained as a result of better precipitation of organic or colloidal matter. It appeared that the optimum amount of lime was that which gave 0.20 alkalinity; and in the secondary defecation it should not be less than 0.5 per cent. Double saturation gave much better results than single, and the juice was clearer, colourless, and more easily filtered.

It seems also that the content of lime salts in the final product was still comparatively high, though less than in 1943, and the question of a certain amount of de-liming may arise. This, however, should not be difficult if phosphates be used as described in the literature. In this connection reference is made to the work of Spengler and co-workers of Andres (*Z. Wirtsch. Zuckerrind. Tech. Sect.*, 1940, 90, 207 and 228). These and others are included in an appended bibliography, most of the references in which are obtained through *Ind. Sacc. Ital.*, 1933-40. It is regretted that the sugar factory at Casalmaggiore had finished its brief season of working before Bonelli's process could be adequately tested.

### Nitrogen Consumption

According to the annual report of Arkman (London), Ltd., resumed after a lapse of some years, world consumption of nitrogen (synthetic, by-product and Chilean) for the year ending June 30, 1947, is estimated at 2,660,000 tons. The demand, which compares with 2,870,000 tons in 1938-39, has been in excess of the supply. A continuing shortage is forecast, with probable price increases.

## WELSH STEEL PLAN "Biggest Ever Carried Out"

THE £50,000,000 scheme for the modernisation and development of the tinplate and sheet steel industry in South Wales has now reached the stage at which decision can be taken on its main features. This was announced by the Minister of Supply in Parliament on Monday, when he described it as "probably the biggest scheme ever carried out."

Making his announcement, Mr. Wilmot said he had considered the recommendations of the Iron and Steel Board and the Government accepted the board's recommendations for the construction by the companies concerned of a new continuous hot strip mill with coke ovens, blast furnaces, steel melting shops and ancillary plant at Margam, near Port Talbot. Further, the Government agreed with the board as to the need for new coal reduction and finishing plant for tinplate and sheet steel. The precise location of this was under consideration.

Replying to a question, the Minister said the scheme would not be in full operation for some three years.

### Official Notices

The Dyestuffs Controller, Board of Trade, 28 Blackfriars Street, Manchester, 3, reminds dyestuffs consumers and merchants that the sale or disposal of any dyestuff is prohibited, except under licence, by the Control of Dyestuffs Order, 1939 (S.R. & O. No. 1431), and Control of Dyestuffs Order, 1940 (S.R. & O. No. 1836).

The Ministry of Supply announces that as from March 28, 1947, the price of tungsten ore of standard grade from Government stocks will be 100s. per unit of WO<sub>3</sub> delivered consumers' works. For special high-grade Scheelite to the following specification—WO<sub>3</sub> minimum 68 per cent, tin maximum 0.60 per cent, arsenic maximum 0.10 per cent, molybdenum maximum 0.10 per cent—the price will be 105s. per unit WO<sub>3</sub> delivered consumers' works.

The Treasury has made the Safeguarding of Industries (Exemption) (No. 2) Order, 1947 (S.R. & O. 1947, No. 649), which exempts styrol (styrolene) and tetrahydronaphthalene from Key Industry Duty until June 30, 1947.

The Board of Trade announces that, as from April 23, 1947, transshipment licences are no longer required for essential oils nor for hydrocarbon oils which are imported and entered with Customs for exportation after transit through the United Kingdom or by way of transshipment.

## INTERNATIONAL PATENTS Agreement with 24 Countries

AGREEMENT has been reached between Great Britain and 24 other countries to regularise international practice regarding patents, trade marks and certain other property rights, the observance of which has been neglected during the war. One of the principal provisions is that any convention period for patents, designs and trade marks which had not expired before the war or arisen after January 1, 1947, will be extended until the end of this year. Proprietors of patents, etc., will be given until June 30, 1948, to satisfy any legal obligation, without penalty, to preserve their rights or to acquire further rights which they would have been entitled to claim had the war not intervened. Renewal of trade marks which expired in the period September 3, 1939—June 30, 1947, will have retroactive effect if renewed before June 30, 1948.

The period from the outbreak of war to June 30 this year will not be taken into account in considering the exploitation of patents or designs, or the refusal or cancellation of them. Compulsory licences or the revocation of patents or designs under Article 5 of the Convention will not be imposed before June 30, 1949. Inventor's and third-party rights arising between September 3, 1939, and January 1, 1946, are to be protected.

The final protocol contains a clause ruling that trade mark rights shall not be prejudiced by infringement caused in the course of importing goods essential for war or for relief of distress caused by war between September 3, 1939, and June 30, 1947.

Parties to the agreement in addition to Great Britain are: Belgium, Brazil, Czechoslovakia, Denmark, Eire, Finland, France, Northern Ireland, Greece, Hungary, Italy, Lebanon, Liechtenstein, Luxembourg, Morocco (French), Netherlands, New Zealand, Poland, Portugal, Rumania, Switzerland, Syria, Tunis and Turkey. The full text is available as published by the Patent Office, 25 Southampton Buildings, London, W.C.2; 1s. net.

### Tin Smelters Fined

A firm of tin smelters, Messrs. Williams Harvey of Bootle, Lancs., were fined £10 for not having electrical equipment properly safeguarded. A workman, it was stated, put his foot on a steel box the plate of which was so rusted that it gave way and his foot touched an electrical condenser carrying 60,000 volts. Although surrounded by blue flashes the man survived to give evidence in court. The firm said every reasonable safety measure had been taken.

# ACTIVATED ALUMINA

## Recent Research in France

WORK on activated alumina, including preparation, use in certain specific cases, and regeneration, has been reported by H. Thibon and co-workers, Cie. Alais, Froges et Camargue, Lab. gén. de l'Alumine, Gardanne, Bouches-du-Rhône. (*Chim. et Ind.*, 1947, 57, 117-125). Active carbon and silica gel have hitherto been regarded as the chief adsorbants for general use, activated alumina being looked upon as a kind of poor relation of silica gel, although, in recent years in the U.S.A. it has come to be preferred above gel in some cases. But as far back as 1933-4 it was already well known in this connection. In *Chem. Markets* for September, 1933, an article on "Alumina gel as adsorbant" described its precipitation with ammonia from an aluminium salt solution; and in the 8th edit. (1934) of Gmelin's *Inorganic Chemistry*, under aluminium, several methods of preparation and examples of its use are given. Two American patents: 1868869 (1928) and 2015593 (1932) of the Aluminium Co., of America (ALCOA) are quoted. In the first of these a temperature of activation between 300° and 800°C., preferably about 350°C., is mentioned. Subsequently ALCOA collaborated with Pittsburg Electrodyer Corporation in the practical development and industrial use of activated alumina. As will be seen the French workers found a much lower temperature of activation sufficient.

### Contraction Under Heat

They prepared the alumina both in powder and in granular form, and in the first place studied the extent of contraction which occurs when the grains are heated to activation temperature. Such contraction is already well known when alumina is calcined at about 1300°C. and transformed into the alpha form. In the present case heating for four hours at 400°C. caused a linear contraction of about 5 per cent (1944 measurements). More recently special observation was made of two particular grains of trihydrate among the largest present. One of these was heated for an hour at 300° and the other at 450°. Contraction in the former—two measurements—was 4.9 and 5.5 per cent, and in the latter 6.5 per cent in both tests. Prolonged heating for several hours produced no further change either in form or dimensions; but the grains previously transparent were now opaque.

One or two generally accepted definitions may be quoted here: (a) adsorption capacity or equilibrium charge is the weight of adsorbed material at saturation per 100 parts by weight of adsorbant; (b) rupture

point, in the special case of water vapour, is the stage at which drying—of air—ceases to be total, as defined by Caillaud and Beaufile (*Chim. et Ind.*, June, 1936). These authors emphasise the fact that the rupture point is retarded in proportion as the last layers of alumina in the column retain the traces of moisture passed by the first layers.

The adsorption of water vapour by alumina may be either static, in an enclosed space, or dynamic, in an air current. In this latter case the special apparatus described by Caillaud et Beaufile (*loc. cit.*) was used, consisting essentially of a dust filter, constant temperature saturator, copper coil and principal absorber, two control absorbers in parallel, gas-meter, and manometer. Mean temperature of the laboratory was 25°C.; that of the saturator thermostat was kept at least 2° below, to avoid condensation of the water vapour in circuit, and 1° above the temperature of the principal absorber thermostat.

### Results of Test

The sample to be tested was placed in the principal absorber, carefully weighed before and after; the two controls were also filled with freshly activated alumina; the temperature adjusted, and a current of air 120 lit./hr. passed through. The two controls were used alternatively each half hour, being weighed each time they were put out of circuit. When the rupture point was reached, as indicated by increase in weight of control absorber in use, the principal absorber was weighed, replaced, and operations continued until there was no further increase in weight. Particle size of the grains of alumina used was a major diameter of 2 to 4 mm. Results may be classified as follows:

1. Effect of activation temperature on adsorption efficiency: The amount of water vapour adsorbed at rupture point (percentage of initial activated alumina) was 15.2 per cent for activation at 310° and 14.4 per cent for activation at 360°. So far therefore as total drying is concerned the lower temperature is to be preferred, and not the 350-360° of the American patents cited *supra*. Time of heating was 30 min. (see 2 below).

2. Effect of time. This was varied from 1 to 1 hour, and the table given indicates that even at 0 adsorption was 14.1 per cent (temp. 310°). But the original text is not entirely clear—it appears that the temperature of 310° was only attained after 2½ hrs. of gradually rising temperature during activation. After 30 min. adsorption was 15.3

per cent and after 60 min. it was 15.1 per cent—measured from the attainment of 310°.

3. Effect of water content or hydration of the activated alumina. Little difference was found in aluminas containing from 8.11 per cent combined water, the adsorption ranging from 15.0-15.6 per cent.

4. Dynamic drying efficiency, with gradually increasing amounts of water vapour introduced into the column. Tables and curves are reproduced from the earlier work of Caillaud and Beaufils (*loc. cit.*) together with comparisons with silica gel taken from the same authors. The usual three grades of the latter—macroporous, microporous, and ultra-microporous—and their adsorption power are quoted. On a weight comparison basis silica gel is shown to be greatly superior, but on a volume basis there appears to be little difference.

### Static Adsorption Tests

Activated alumina in powder form was used for the static adsorption tests, and was prepared by heating aluminium trihydrate in a suitable furnace. Temperature of activation ranged from 265° to 350°, and loss of moisture is shown as function of time and temperature as follows:

Temp. of activation	Per cent calcination loss after			
	1 hr.	2 hrs.	3 hrs.	7 hrs.
265°	26.3	18.6	16.0	12.6
300°	19.4	14.5	10.3	9.0
325°	15.7	10.3	9.5	8.5
350°	9.2	8.1	7.9	7.5

The saturation capacity of aluminas thus activated was tested by placing samples in flat flasks which were covered only at the moment of weighing. These were put in a Scheibler dryer containing water to provide a saturated atmosphere, and the whole left in a room at 18°C. for a fortnight. The flasks were periodically weighed and the operation was considered complete when there was no further increase in weight. The moisture retained reckoned as percentage of the original activated alumina, was as follows:

Temp. of activation	per cent adsorption capacity at 18° C obtained after an activation of			
	1 hr.	2 hrs.	3 hrs.	7 hrs.
265°	5.7	12.0	13.2	16.5
300°	13.0	15.5	19.0	19.5
325°	17.0	22.1	22.0	22.0
350°	22.7	23.0	22.9	22.9

It will be seen that a temperature of 325°C. is required for activation without air current. At 350°C. static activation is complete in 1 hr. In comparing these figures with those obtained in the dynamic series with granular alumina it is thought that there is no very marked difference. It is pointed out, too, that alumina saturated with moisture may be regenerated by heating at 150°; unless it has been left wet for some considerable time (several weeks) when some form of chemical adsorption may reinforce

that which is purely physical, and a temperature of 3000°C. is required. (Cf. Travers on Physical adsorption and chemical adsorption, *Chim. et. Ind. Technologic*, 1944).

In studying the efficiency of alumina for adsorbing chlorinated organic solvent vapours, namely, tetrachlorethane, di- and trichlorethylene, comparison was made with silica gel, based on work done in 1934 and covering only saturation capacity without reference to rupture points. The solvent vapours were carried in an air current at 90 lit./hr. On a weight basis again the silica gel proved markedly superior, but on a volume basis the authors think there is little to choose. They note the appearance of capillary condensation, *i.e.*, liquefaction of the vapour in the pores of the adsorbent.

In some further tests with Congo Red in aqueous solution and with arsenious anhydride ( $As_2O_3$ ) in alkaline solution it was found that maximum adsorption occurred with an activation period which was so much the shorter as the temperature of activation is higher—up to 350°C. The hope that either or both of these substances would provide a more commodious and quicker method of determining adsorption capacity was not apparently realised. With iodine in potassium iodide solution adsorption was too slow for this method to be of any practical value in such determinations.

In work on the regeneration of activated alumina a powdered Bayer product was used, activated by heating for two hours at 350°C., and employed for adsorption of water vapour. Samples were placed in a cloche in a saturated atmosphere at 22°C., and results are tabulated, showing what the authors term the kinetics of hydration and rehydration of activated alumina new and regenerated up to eight times, the period of exposure to the saturated air ranging from 0 to 160 hours. It was found that the samples could be satisfactorily regenerated several times by heating for one hour at 155°C.

### Changes in Structure

Finally, in discussing the nature of activated alumina, the authors review the changes in structure which accompany activation of dehydration. They distinguish between the external and the internal surface, the latter being related to porosity, and compare adsorption powers—for Congo Red—of an unactivated alumina of varying fineness and external surface; and, as expected, adsorption increases with area of external surface ( $cm.^2/gr.$ ). When activated there is an increase in internal surface area through formation of pores accompanied by destruction or modification of original crystalline form or lattice and removal of water of constitution, as well as changes in molecular structure of



the hydrargillite. As stated long ago in Gmelin (*loc. cit.*) the trihydrate when progressively heated is transformed first into boehmite at about  $200^{\circ}$  ( $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ), then into gamma-alumina from  $300^{\circ}$  upwards, and finally into alpha-alumina or corundum at about  $1000^{\circ}$ . Activated alumina, at least in theory, should consist of a mixture of boehmite and gamma alumina in about equal parts. An X-ray study of samples submitted to the Physics Laboratory of the

company (Alais, Froges, etc.) at Chambéry, using the Debye-Scherrer method, revealed the same structure diagram as boehmite of orthorhombic crystalline form. Reference is made to some investigations by Belestki (*Legkie Met.*, 1936, Feb.) and R. Michaud (*Bull. Soc. franc. Phys.*, 1943, May) on the changes in crystal structure of alumina under varying temperature and other conditions, and the relation of these to adsorption capacity.

## Future of the German Coal-Oil Industry

IT is now expected that in three or four months' time the synthetic fatty acid works at Witten will again receive paraffinous raw material ("Gnatsch") from synthetic coal-oil plants. Two of the latter, those of Gewerkschaft Viktor or corindon at about  $1000^{\circ}$ . Activated alumina, at least in theory, should consist of a mixture of boehmite and gamma alumina in about equal parts. An X-ray study of samples submitted to the Physics Laboratory of the

### Use of Hydrogenation Plant

Meanwhile a scheme has been presented to the Military Government providing for the use of the hydrogenation plant of Gelsenberg Benzin A.G. at Gelsenkirchen for the hydrogenation of imported oil refinery cracking residues. For every five tons of bituminous coal which would otherwise be required for this plant 1.27 tons of coal and 1.15 tons of residues obtained in the course of cracking operations by foreign oil refineries would be used, with a 40 per cent saving of the foreign currency needed to import an equivalent amount of petrol. It is claimed that after war damage repairs, scheduled to take about six months provided the necessary construction materials can be obtained, a monthly output of 11,000 tons of motor spirit, 1,100 tons of fuel gas, and 1,100 tons of hydrocarbons for the production of synthetic rubber, as well as sulphur and sulphuric acid could be procured.

The raw material intake would include 13,800 tons oil residues, 2,500 tons of coke (for hydrogen production), 10,000 tons of coal (of an inferior type) and 6,200,000 cubic metres of hydrogen (to be supplied by the Chemical Works at Huels through an existing pipeline). The methane gases obtained as a by-product of the hydrogenation process would be disposed of to the Huels works for the manufacture of buna and solvents.

An earlier plan provided for the resumption of hydrogenation at the plant of Rheinische Braunkohlenkraftstoff A.G. at Wesseling, but this has been abandoned in favour of an alternative scheme to use the plant for the synthesis of ammonia. While part of the Wesseling plant is even now being

used for the production of methanol (at a monthly output rate of 1500 tons), other parts of the works could, it is claimed, be converted to the ammonia synthesis without great difficulty or expense. A monthly production of 3000 tons of nitrogen is aimed at, which could be shipped to the works at Hoechst for processing into compound fertiliser. For this output 7500 tons of lignite briquettes and 7000 kW would be required.

Substantial progress has recently been reported by the artificial fibre industry. The J.P. Bemberg A.G. has received permission to resume operations in part of the rayon plant at Wuppertal-Oberbarmen. The works of Spinnfaser A.G., Kassel-Betterhausen, have been repaired, so that a daily production of approximately 50 tons a day could be procured if the necessary coal and raw materials can be obtained; the pre-war capacity exceeded 100 tons daily. Rheinische Zellwolle A.G., Siegburg, also reports considerable progress in clearance and repair work, with the result that the plant could now resume production on half the pre-war scale. It is hoped that the latest negotiations with the Scandinavian countries will permit the resumption of cellulose imports.

### More Mexican Plastics

With the value of output increasing from approximately \$300,000 in the first half of the year to \$500,000 in the second half, and with plants working at capacity by the end of the year, indications are that the Mexican plastics industry, with firm prices prevailing and a steady demand, experienced a successful year in 1946. Although almost all of the country's plants are small, the advances made are impressive, especially since the industry in general did not begin production operations until 1944-45.

In the remainder of 1947, Mexican plastics manufacturers hope to more than double the 1946 totals and achieve a \$1,000,000 or more output for the year. However, as lower prices are expected, a tentative value of only \$1,200,000 has been estimated for the 1947 production.

## SWEDISH IMPORTS : LICENCE-FREE GOODS

**A**CCORDING to the *Board of Trade Journal*, which gives details of the new system of import licensing in Sweden, the following are included in a "Free List" of goods not requiring Import Licences.

Mineral and fossil products: asbestos, bauxite, cryolite, even artificial; manganese, gypsum, mica, emery, ores even in bricks. Peat briquettes, other fuel not separately mentioned; peat dust and peat turf litter, carbolic acid and cresol, lubricating oils, also transformer oil.

Chemical products and pharmaceutical goods; colours and varnishes, perfume products; soap, candles and other products of oil, fat or wax; glue, gum and gelatine; explosives; fertilisers: benzoic acid and salicylic acid, glycerine, potassium and sodium xanthogenates, lead oxide, potassium chloride, calcium chloride; oxides, hydrates, fluorides, chlorides, chlorates, bromides, iodides, sulphates, sulphites, hyposulphites, nitrates, carbonates, borates, perborates, chromates, molybdates, permanganates, formates, tartrates, benzoates and carbides of metals or ammonium, not classified under any other number; other kinds than those under Statistical Nos. 493 : 1 to 500 and 502 to 512.

Wood spirit (methanol), phthalic acid esters not separately mentioned; acetophenolisatine, anevrin hydrochloride, succinic acid, diethylamine, diethylaminoethanol, diethylene, diethylsulphate, diphenolisatine, dihydrocholic acid, dichlorhydrine, dimethylamine dimethylaniline, dimethylsulphate, ergosterine, ethylene diamine, ethylene chlorhydrine, phenyl hydrazine, glutaminic acid hydrochloride, histidine, calcium lactate, calcium rhodanide, capronic acid, quinine, chlordinitrobenzol, lactoflavine, lithium citrate, mandelic acid, metacresotinic acid, methylacetamide, monomethylamine, sodium, amide, sodium anhydromethylene disphosphate, sodium glutamate, sodium orthoiodohippurate, sodium salicylate, nicotinic acid, nicotinic acid amide, orthoxyquinoline, paramethoxybenzaldehyde, para-oxybenzoic acid methyl ester, para-oxybenzoic acid ethyl ester, para-oxybenzoic acid propyl ester, para-toluol-sulphonic acid methyl ester, para-toluol-sulphonic acid ethyl ester and sulphosalicylic acid.

Chemical preparations, not separately mentioned, except butyl alcohol and vitamin-A oils.

Tanning extracts, colours, varnishes, etc. Lampblack and similar black colouring substances, not separately mentioned. Red lead.

Soap, candles, etc., machine- and cart-grease; lubricating oil, of a mixture of fat oil and mineral oil. Other greases, not

separately mentioned, in which oil or fat are used.

Explosives, fireworks and matches; percussion caps.

Machines, apparatus, and implements, not electric.

Boilers, heaters, not separately mentioned, also containers and cookers, with machine regulators, all items if made mainly of malleable iron; also manufactures of rolled iron sheet for boilers. Furnaces for steam-boilers and gas works; also grates for such apparatus. Economisers and parts thereof. Condensers. Ovens for industrial purposes, also forges and bellows. Rolling mills for the metal industries, steam, pneumatic and spring hammers, riveting and wire-drawing machines, nail, horseshoe and forging machines; punching, cutting, curving and straightening machines, pressing machines including presses, not separately mentioned; also sheet-metal-workers, copper-smiths and tin-smiths machines, not mentioned above. Shears for flat or profiled iron, also machines for punching, curving and straightening, each weighing more than 8000 kilog.; also pressing machines and metal-working presses, weighing more than 30,000 kilog. each.

Instruments, except musical instruments, and weighing machines. Thermometers, water-meters, manometers and vacuum meters and parts thereof; gas meters weighing at most 100 kilog. each; electricity meters and other electrical measuring instruments and parts thereof.

Phenol-formaldehyde synthetic resins and other artificial resins for pressing, capable of hardening or hardened, also manufactures thereof, not separately mentioned; even containing paper, textile materials, etc.

It has been ascertained that import licences will be issued immediately for certain essential goods, e.g., the more important raw materials, semi-manufactured and finished goods, such as important chemicals, textiles, machines, etc.

Our attention has been drawn to the fact that imports of chemicals from America have to be supported by the necessary import licence issued by the Board of Trade, and that these licences are issued only after complete investigation and support of the case for importation given.

The Minister of Food has announced an agreement with the Scotch Whisky Association by which permission will be given to the distillers to buy 50,000 tons of barley out of the 1946 crop and, subject to harvest, a further 75,000 tons in the autumn out of the 1947 crop. In return, the Association has agreed to raise to 75 per cent the proportion of its current releases for export.





## A CHEMIST'S

### BOOKSHELF

**Poisons: Their Chemical Identification and Emergency Treatment.** Vincent J. Brookes and Hubert N. Alyea. D. van Nostrand Co., Inc. Macmillan and Co., Ltd. pp.xiv + 209. 16s. 6d.

This book reached England about the same time as the report of a lecture on industrial hygiene and safety, by the recipient of the 1946 Pittsburgh Award of the American Chemical Society. In this address Dr. Yant stated: "... there is some indication that the chemical profession is becoming aware of a need for safety, perhaps by the inclusion of these subjects in texts or lectures. I would go even further and recommend a course of instruction in safety and chemical toxicology as a requirement for graduation." At first sight it appeared that the book by Brookes and Alyea would offer the necessary instruction, but this is hardly the case.

In fairness to the curiously-met authors, a police sergeant and an assistant professor of chemistry, it should be explained that the primary purpose of the book is to assist the police officer in recognising the symptoms of poisoning. The authors have had an extremely difficult job, and it may be that in this primary purpose they have been largely successful. However, it is to be feared that the average police sergeant, even if trained in the techniques described in the book, would have a very "hit-or-miss" success in rendering first aid after applying the chemical tests described.

It is undoubtedly true that recognition of the existence and the precise nature of poisoning is more in the line of the man with a medical training. Detailed analysis of poisons may be the job of the specialist analytical chemist, but this aspect of the problem is not meant, and is not considered by the authors, to be within the abilities of the police officer. Therefore it is noteworthy that in almost every case one reads that the first instruction for treatment is "summon a physician." The first instruction for recognition of the poison, almost as implicitly stated, is to ascertain the source of the poison and to read the label on the container. The reviewer feels that for the amateur these two instructions would suffice, and the elaborate reagent kit, containing seventy-three reagents, which the officer presumably carries round with him, would seem less reliable, even after the officer has received "expert instruction for several weeks by a trained toxicologist or chemist."

To write chemistry for non-chemists is most difficult, and Professor Alyea has obviously met the normal problems and in some instances has failed to surmount them. If, (p. 14) it is necessary to give the reader a list of symbols for the commoner chemical elements and the information that "atoms unite to form compounds," and (p. 15) that "carbonic acid ( $H_2CO_3$ ) forms the fizz in soda water," it is likely that the same hypothetical reader will be left cold by the statements (p. 37) that nitric acid "is prepared industrially by the catalytic oxidation of ammonia" or (p. 38) that oxalic acid crystals "are so stable that they are used as an oxidation-reduction standard in analytical chemistry." One speculates also about the reasons for including in the glossary the terms "characteristic," "contracted," "deceased," "flexible," "inflammable," "perspiration," "solid," "variable," to choose almost at random.

The book is not, however, by any means without value. It offers, excellently tabulated, a list of poisons and their antidotes, about which the average worker in a chemical laboratory is unfortunately usually rather vague. There is also a good section on industrial hazards, their toxic concentration, and the treatment for poisoning by such substances. The chapter on food, plant, snake and spider poisoning is not particularly applicable to this country. But there is also a useful section on special treatments, in particular artificial respiration and treatment for shock.

Any laboratory which has not yet equipped itself with a simple reference book on poisons and their treatment could do worse than to place this book in a readily accessible location, always provided that it expects its occupants not to form themselves into a corps of private investigators but to use the book wisely in emergency and to study it occasionally in tranquillity.

CECIL L. WILSON.

#### **Aluminium Scrap Imports**

The Minister of Supply announces that, with the agreement of the President of the Board of Trade, imports of aluminium scrap and aluminium alloy scrap will now be allowed. Imports will be subject to individual licencing and applications should be submitted in the usual manner to the Import Licencing Department, Board of Trade, 189 Regent Street, London, W.1.

## Parliamentary Topics

**T**HE allocation of urea and butyl alcohol was the subject of some discussion in the House of Commons between the President of the Board of Trade and Mr. Wilkes, who asked why the Board of Trade refused to disclose to industrialists the basis of the present allocation; why British Resin Products, Ltd., was receiving greater quantities than development area projects such as British Paints, Ltd., and, as greater quantities of urea had been produced than anticipated, whether this increased production had been allocated to firms within the development areas.

Sir Stafford Cripps explained that when a shortage of urea and butyl alcohol developed early in 1946 monthly allocations to consumers were stabilised at the quantities they were then receiving. This basis of allocation was known to synthetic resin manufacturers generally. There was a small increase in urea production in the second half of 1946 but it was insufficient to offset the drain on stocks. Allocations remained constant until early in 1947 when, owing to the exhaustion of stocks, all consumers suffered a pro rata reduction except for firms taking only small quantities.

Mr. Wilkes submitted that this firm, outside the development areas, was not, so far as was known, a firm which used these substances in prewar days; and, according to information, they apparently now received a larger allocation than any firm within the development areas who had recently started up.

Reiterating that stabilisation took place in 1946, Sir Stafford said British Paints, Ltd., had not suffered any reduction because it was a small firm.

Mr. Wilkes further asked the President whether, in view of the fact that the price of home produced urea was £31 per ton as compared with £76 per ton for imported German urea, he would create, as previously done in the case of phenol, a urea pool of both imported and home produced urea at a uniform price level to spread the cost; and whether he was aware that in default of such a pool new development area projects would be placed at a disadvantage in competing with prewar users outside the development areas who, on the basis of their prewar urea consumption, received much larger allocations of home produced urea at the lower price.

Sir Stafford: A small consignment of urea has arrived from Germany after protracted negotiations. There is at present no certainty as to continuing supplies but, if the position improves in this respect, I will certainly consider whether some pooling arrangement could be reached.

Mr. Wilkes asked the Minister to bear in

mind that firms within the development areas had to refuse allocations of this German exported urea because it was not at competitive prices; and Sir Stafford replied that it was expensive compared with others, but with the shortage of urea they were trying to get everything they could.

### Sulphate of Ammonia

Supply and distribution of sulphate of ammonia are planned for the 12 months, July to June. For the period July 1, 1946, to June 30, 1947, exports under International Emergency Food Council allocation should be about 266,000 tons. Shipment of 194,000 tons has already been made and the balance will depend upon availability.—President of the Board of Trade.

## NEXT WEEK'S EVENTS

### MONDAY, APRIL 28

**The Chemical Society.** (Newcastle and Durham Section). Chemistry Lecture Theatre, King's College, Newcastle-upon-Tyne, 5 p.m. Meeting for the reading of original papers.

### TUESDAY, APRIL 29

**Society of Instrument Technology.** National Physical Laboratory, Teddington. 7 p.m. Mr. J. A. Hall: "Molten Metal Temperature Measurement."

### WEDNESDAY, APRIL 30

**The Institute of Welding.** Great George Street, Westminster, London, S.W.1, 6 p.m. Dr. Maurice Cook and Dr. Edwin Davis: "The Welding of Copper and the Copper-Rich Alloys."

### THURSDAY, MAY 1

**The Chemical Society** (London Section). Burlington House, Piccadilly, London, W.1. 7.30 p.m. Mr. C. W. Scaife: "Nitration of Olefins," Mr. A. Lambert: "Reactions of the Nitro-paraffins," Mr. R. L. Heath: "Addition Reactions of Nitro-olefins," and Mr. G. D. Buckley: "Formation and Reduction of 2- and 3-nitro-alkyl Cyanides."

**International Society of Leather Trades Chemists** (British Section, Northampton Group). College of Technology, St. George's Avenue, Northampton, 2.30 p.m. Dr. J. A. Lovren: "Fish Oils."

### FRIDAY, MAY 2

**Society of Public Analysts and other Analytical Chemists.** (Physical Methods Group). Chemistry Lecture Theatre, King's College, Newcastle-upon-Tyne, 6 p.m. Dr. C. E. Ransley: "Gas Analysis at Low Pressures," Mr. J. H. D. Hooper: "The Analysis of Hydrocarbon Gases by Low Temperature Distillation," and Mr. W. J. Gooderham: "A New Apparatus for Gas Analysis by the Soap Film Method."

## Overseas News Items

**Oil Search in Australia.**—The Australian Government, in conjunction with Zinc Corporation and Field Oil Corporation of Los Angeles, have begun a search for oil in the Kimberley area.

**Chemicals on Brazil's Control List.**—Among the chemical materials still in short supply in Brazil are caustic soda, potash, rosin, and Chilean nitrate and these commodities, as a result, are among the 20 items still remaining on the control list of Brazil's Federal Foreign Trade Council.

**Rise in Malayan Rubber Exports.**—There was an increase of nearly 22,000 tons in rubber exports from Malaya during March, by comparison with February, the figures being 91,367 tons as against 69,577. Destinations were as follow: United States 34,524 tons; United Kingdom 16,140; Canada 8462; Argentina 7376; Hongkong 4931; France 3898; Soviet 3750; South Africa 1654; Australia 1519; Czechoslovakia 1352; Sweden 1363.

**New Sulphuric Acid Plant in Mexico.**—In January of this year a new sulphuric acid plant, with a daily capacity of 50 metric tons, began operations in San Luis Potosi, Mexico. The plant's entire output is destined for the local fertiliser factory, Guanosa y Fertilizantes, S.A. The contact process is employed by the plant in manufacturing the sulphuric acid. Eventually it is hoped that waste sinter plant gases will become the raw material source.

**Ten Killed in Fireworks Factory.**—Ten workmen were killed and several others reported trapped in a fireworks factory explosion at Clinton, Missouri, on April 2. Twenty-five persons were in the building at the time of the explosion, which was followed by a fire. The bodies recovered were so badly burned that they could not be identified. Police officers said that the holocaust created such intense heat that it was impossible to get within 100 yards of the building.

**Argentina Sets Calcium Carbide Quotas.**—The 1947 import quota in Argentina for calcium carbide has been fixed at 11,000,000 kilograms by the Argentine Department of Industry and Commerce. The quota thus established will be apportioned among the usual importers of this product. In order to obtain an import permit, the importer must present a certificate, issued by the General Bureau of Military Manufactures, showing that he has already acquired his proportional share of the domestic production of calcium carbide.

**Argentine Coal and Iron.**—Good quality coal in substantial quantities has been obtained from the mine at Rio Turbio in the territory of Santa Cruz, where there is stated to be a coal seam estimated to be capable of yielding 100 million tons, according to an Argentine Government announcement. The Government expects the deposits of iron in the provinces of Jujuy and Salta will produce about 100 million tons of iron ore.

**Record Bauxite Output in U.S.**—Domestic production of a third of a million tons of crude bauxite in the third quarter of 1946 was the greatest in the United States since the end of 1944 and exceeded that of any quarter prior to 1942. Output, partly estimated, was 341,079 long tons (289,917 tons dried equivalent) in the third quarter, a gain of 13 per cent over the 303,163 tons (257,689 tons dried equivalent) produced in the second quarter.

**Austrian Hermann-Goering Works Busy.**—Three blast furnaces have in recent months been in operation in the Hermann-Goering works, Linz, Austria, producing an aggregate of about 500,000 tons of pig iron. Large export orders are reported to have been received for a variety of products, but delivery depends on an improvement in the coal situation. It is assumed, however, that these works will in future play an important part in the country's economy.

**Czech Iron and Steel Output.**—Czechoslovakia produced last year 961,000 metric tons of pig iron and 1,668,000 tons of crude steel, as compared with 1,674,000 tons and 2,318,000 tons respectively in 1937. The target figures for the current year have been set at 1,340,000 and 2,200,000 tons, necessitating an increase in output of about 40 per cent and 32 per cent respectively. Transport difficulties are mainly blamed for the slow recovery of this important sector of the country's heavy industry.

**Monsanto Chemical to Get Betatron.**—Shipment of the first 100,000,000-electron-volt betatron to be built on a commercial basis is now underway, according to the General Electric Company (U.S.A.). The machine is being built by G.E.'s General Engineering and Consulting Laboratory, under the engineering supervision of Dr. G. W. Dunlap, for the Clinton Laboratories, operated at Oak Ridge, Tenn., by the Monsanto Chemical Company for the Atomic Energy Commission. G.E. is also building a similar betatron for the University of Chicago, where it will be used for nuclear research.

## Personal

MR. E. BASIL GREEN has been appointed joint managing director of Doulton & Co., Ltd.

MR. B. J. DAVIS and MR. C. H. AIKMAN DANKS have joined the board of Aikman (London), Ltd.

MR. G. H. LATHAM is the new president of the British Iron and Steel Research Association, having succeeded Sir James Lithgow.

MR. JOHN MACASKILL, formerly secretary of Fabon, the Government organisation concerned with fats and glues, and now with British Glues and Chemicals, Ltd., has been appointed a director of the latter company.

DR. J. G. KING, Director of the Gas Research Board, accompanied by Mrs. King, leaves for America in the *Mauretania* to-day and will be away for some two months, visiting the main technical bodies connected with the gas industry in the United States.

MR. W. A. RUNDLE, of A. Gallenkamp & Co., Ltd., was at the recent annual meeting of the British Laboratory Ware Association, Ltd., elected chairman for the ensuing year upon the retirement of MR. NORMAN TREPTE, of W. and J. George & Becker, Ltd., who had held office for the past three years.

DR. HAROLD HOLLINGS, controller of research with the Gas, Light and Coke Co., has been appointed chairman of the council of the Gas Research Board, in succession to Dr. E. V. Evans, who has resigned. Viscount Falmouth has been re-elected president.

MR. G. G. BARNES has been appointed by the Minister of Food to be a Government director of the British Sugar Corporation, filling the vacancy caused by the death of Sir Louis Kershaw. Mr. Barnes, who recently retired from the Ministry of Food, was secretary of the Sugar Commission before the war and assistant secretary in the Sugar Division of the Ministry of Food from the outbreak of war until his retirement.

DR. JAMES IRVIN HOFFMAN, of the U.S. National Bureau of Standards, an outstanding analytical chemist well known for his work on the chemistry of the rarer elements and the chemical analysis of iron, steel, and various minerals such as phosphate rock, bauxite, and fluorspar, has been awarded the 1946 Hillebrand prize for significant contributions to chemical science by the Chemical Society of Washington, D.C. The Hillebrand prize, which is awarded annually, was established in 1925 in honour of William Francis Hillebrand, chief chemist of the National Bureau of Standards from 1908 to 1925. Dr. Hoffman's specific achievements were listed as the

determination of the atomic weights of aluminium and gallium, the development of a hydrochloric acid extraction process for the production of alumina from clay, and



Dr. J. I.  
Hoffman

the development of an ether extraction process for the purification of uranium oxide, by which was removed the greater part of the difficulties encountered by the Manhattan District in securing pure materials for the production of the uranium metal.

## Obituary

SEÑOR SIMON IRTURBI PATINO, Bolivian multi-millionaire, and known as "tin king," has died in Buenos Aires at the age of 87. He was president of Consolidated Tin Smelters, Ltd., General Tin Investments, Ltd., and Patino Mines and Enterprises Consolidated.

DR. JOHN DOWNIE FALCONER, geologist and geographer, who died on April 16, aged 70, went out to Northern Nigeria in his younger days as principal of the Mineral Survey and from 1918 until 1927 was Director of Geological Survey for Nigeria. From 1928 to 1934 he was geologist to the Uruguan republic. Of many books and papers, his most important were those based on his specialised knowledge of the Nigerian tin-fields.

MR. JOHN FLOYD BOTTOMLEY, senior life director of J. C. Bottomley & Emerson, Ltd., Brookfoot Works, Brighouse, Yorks., manufacturers of paints, fertilisers, and aniline dyestuffs, died at his home on April 12, aged 79. Mr. Bottomley was the son of John Carr Bottomley, who founded the business of J. C. Bottomley, drysalter, in 1857. He spent 10 years in wool spinning at Dewsbury before entering the chemical trade on the death of his father in 1898. Mr. Emerson joined him in 1919 and the limited company under its present title was formed.

## Home News Items

**Trade Mission in Moscow.**—A British trade mission, led by the Secretary for Overseas Trade, Mr. Harold Wilson, has arrived in Moscow.

**Lack of Fertiliser Chemicals.**—Chemicals urgently required for fertiliser manufacturing purposes are now so scarce that manufacturers are experiencing the greatest difficulty in continuing production. According to Central Farmers, Ltd., no potash has been available to fertiliser compounders for several weeks, while prospects for sulphate of ammonia are also poor. Instead of improvement in the position there has been a steady deterioration, states Mr. Archibald Dryburgh, chairman of this company.

**Scottish D.S.I.R.**—A Scottish section of the Department of Scientific and Industrial Research is to be established in Edinburgh, and it is hoped to have the station operating by July under the control of a senior research scientist. The scope or terms of reference of the new section have not yet been indicated. There are at present some few units of D.S.I.R. operating on specialised subjects in Scotland—for instance, fish and agricultural machinery—but no central reference section has hitherto existed.

**Electricity Charges Illegal?**—The Engineering Industries Association, representing 3000 British firms, recently protested to the Electricity Commissioners because several of its members were charged full tariff rates for electricity during February when, owing to the fuel shut-down, no current was supplied for three weeks. The Fuel Minister has twice stated in the House of Commons that reduction in charges could not be made. Legal opinion sought by the E.I.A. suggests that the case for exacting the full charge could not be substantiated at law.

**Trade With Belgo-Luxembourg Union.**—Trade discussions in Brussels between an official United Kingdom delegation representing the Board of Trade and the Treasury and representatives of the Belgian and Luxembourg governments have been concluded. The discussions have led to agreement over a wide range of matters affecting the trade between the United Kingdom and the Belgo-Luxembourg Economic Union. The delegations agreed on measures to facilitate substantial exports to the United Kingdom from Belgium and Luxembourg of a wide range of goods, including steel and steel products and other metals, consumer goods, and horticultural produce.

**Heligoland "Blown Up."**—Seven thousand tons of explosives were buried on the island of Heligoland and were detonated by radio and electric cable from vessels nine miles out to sea in order to destroy this former German fortress.

**Imperial Institute Report.**—The wide field covered is reflected in the Imperial Institute's annual report (1946) just published. Chiefly interesting are the reports of the analyses and trials which the scientific departments have carried out on rubber, mineral resources and plant and animal products in the quest of new sources of essential materials.

**I.C.I. Veterans.**—Dr. J. W. McDavid (chairman of the explosives division of Imperial Chemical Industries, Ltd.) presented long service awards to 20 officials and employees at the company's explosives factory at Haswell. Recipients with the longest service were Mr. W. R. Moore, now manager of the satellite factory (35 years) and Mr. S. Berry, chief clerk (23 years).

**World Trade Organisation.**—After preliminary delays, the preparatory committee of the World Trade Organisation meeting in Geneva was expected this week to open tariff negotiations. Following reports by the various delegations as to which countries are ready for the first stage, submission of requests for concessions, and which are ready for the second stage, statement of concessions offered, bilateral meetings will be arranged. More important will be the talks between the major powers, expected to begin before the end of this week.

**F.B.I. and Economic Outlook.**—A joint statement issued by the Federation of British Industries and the Board of Trade on April 17 says: "An interview took place yesterday between Sir Stafford Cripps and representatives of the Federation of British Industries, when the interim statement recently issued by the federation dealing with the economic outlook [THE CHEMICAL AGE, April 5, p.403] was fully discussed. Sir Stafford indicated that many of the recommendations made had been adopted, and gave some preliminary indication of the Government's intentions with regard to the formation of the new industrial planning board through which it is hoped to seek the views of industry in the formative stages of the work of the Chief Planner. Further discussions on this will be arranged shortly."

## Commercial Intelligence

The following are taken from printed reports, but we cannot be responsible for errors that may occur.

### Winding-Up Petition

**TANNIN & CHEMICAL TRADE, LTD.** (W.U.P., 26/4/47.) A petition for the winding-up of this company by the High Court of Justice was on April 3 presented by Watford Chemical Co., Ltd., 17 Half Moon Street, London, W.1, chemical manufacturers, and was to be heard at the Royal Courts of Justice, London, on April 21.

## Company News

A total profit of £430,437 (£454,807) for the year to March 31 is announced by **Bryant and May, Ltd.**

The usual interim dividend of  $7\frac{1}{2}$  per cent has been declared by **Murex, Ltd.**, in respect of the year to June 30, 1947.

A profit of £153,370 (£106,070) for 1946 is reported by **Manganese Bronze and Brass Co., Ltd.**, and a final dividend of  $22\frac{1}{2}$  per cent ( $17\frac{1}{2}$ ) is payable.

A net profit of \$29,681,352 for 1946 was made by the **International Nickel Co. of Canada, Ltd.**, equal to \$1.90 a share on the company's common stock.

**Aluminium Company of America** in 1946 had a net profit of nearly \$24 million, according to the company's annual report, equivalent to \$3.70 a share of common stock, compared with 2.85 a share for 1945.

**Aluminium Company of Canada, Ltd.**, and subsidiaries, for the year ending December 31, 1946, reported a consolidated net profit of \$11,581,237, or \$7.30 a share, after preferred dividends.

**The American Cyanamid Company** and subsidiaries had a net profit of \$8,692,881, equal to \$3.55 a common share, which compares with \$6,213,054, or \$2.34 a share, in the preceding year.

**The Standard Chemical Company, Limited**, has declared a dividend of 10 cents a share on the common stock, payable June 1, to record April 30. This is the first dividend on the common since a five-for-one split in mid-1945.

Steady increase in output and highly satisfactory results for all works of the group were referred to by the chairman, Sir Ronald W. Matthews, at the annual meeting of **General Refractories, Ltd.** Among the year's important projects which he described was the completion of a new plant at the works of the group's subsidiary, **Henry Foster and Co., Ltd.**, for production of carbon blacks for the blast furnace industry—a new development in this country, he said. [See also *THE CHEMICAL AGE*, April 12, p.450].

## New Companies Registered

**H. W. Kay & Co., Ltd.** (432,328).—Private company. Capital £10,000 in £1 shares. Manufacturers of chemicals, fine chemicals and chemical products, etc. Directors: H. and A. Kay. Registered office: 1-3 New Street, Manchester, 10.

**Danco Export and Import Ltd.** (432,835).—Private company. Capital £1,000 in £1 shares. Manufacturers etc., of chemicals, colourings, etc. Subscribers: W. Lindsay, F. King. Registered office: 43, Church Road, Hove.

**Vistula Export and Import Co., Ltd.** (432,805).—Private company. Capital £500 in £1 shares. Oil and chemical brokers, merchants and factors, waste oil dealers, etc. Subscribers: N. Wood and P. Wood. Registered office: 45, Cromwell Road, London, S.W.7.

**Irish Mouldex, Ltd.** (11,845).—Private company. Capital £250,000 in £1 shares. Manufacturers, refiners and reclaimers of rubber, plastics, etc. Directors: J. Stafford, jr., Cromwells Fort, Wexford, Eire, W. Stafford, J. Lewis, M.P.; S. Lewis, J. Harvey.

**Northern Glass and Instruments Ltd.** (432,354).—Private company. Capital £5000 in £1 shares. Manufacturers of chemicals, gases, glassware and laboratory requisites, etc. Directors: G. Adamson, H. Curry, P. Joram, W. Stoker, J. Brown. Registered office: 25 John Street, Sunderland.

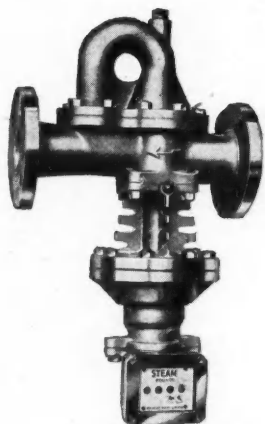
**Birmingham Pelleting Co., Ltd.** (432,297).—Private company. Capital £3000 in £1 shares. Manufacturers of compounds for the plastic, erinoid, bakelite and allied traders and for pharmaceutical and chemical purposes, etc. Directors: L. Ballard, J. Gray. Registered office: 51 Bell Street, Tipton.

**International Minerals and Chemicals, Ltd.** (432,339).—Private company. Capital £5000 in £1 shares. To act as agents in Great Britain or elsewhere and to carry on business of warehousemen, ship brokers, manufacturers of ores, metals, minerals, chemicals and chemical products, fertilisers and fertilising products, vegetable products, etc. Directors: L. Ware, R. Risch, B. Voss, O.B.E. Registered office: Fenwick Buildings, 292 High Holborn, London, W.C.1.

## Chemical and Allied Stocks and Shares

**B**USINESS in stock markets has centred on industrial shares, though buying was selective and based mainly on calculations of the extent to which some companies seem likely to benefit from the abolition of E.P.T.





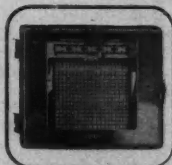
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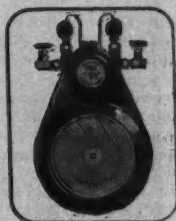
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British Funds, after easing, firmed up, and, generally, prices in most sections have tended to move higher with buyers predominating, although best prices were not held.

Awaiting the full results, Imperial Chemical moved up to 48s. 1½d., at which the yield on the increased 10 per cent payment works out at 4½ per cent. Market calculations as to E.P.T. abolition benefits drew renewed attention to Turner & Newall, which rose afresh to 91s. British Oxygen were 101s. 10½d.; British Aluminium 47s. 9d.; Borax Consolidated 60s., and United Molasses 57s. 10½d. Lever and Unilever improved to 53s. 6d., and Goodlass Wall were 34s. 6d. Elsewhere W. J. Bush, which remained firmly held, were 93s. 9d. B. Laporte were also quoted at this level; and Fisons changed hands around 61s. 9d. Glaxo Laboratories were £21½; and, on E.P.T. abolition benefits, British Drug Houses have advanced to 67s. 6d., while Beechams deferred showed strength at 29s. 6d., and Aspro ordinary also moved up to 50s. 6d. Amalgamated Metal rose to 21s. 7½d. in response to hopes that the London metal markets may re-open this year. Imperial Smelting were 20s. 6d., and in other directions, Allied Iron-founders moved up to 65s. Iron and steel shares were favoured in view of their attractive yields and the benefits the companies may in many cases derive from the abolition of E.P.T. Guest Keen were 47s. 9d.; United Steel 27s.; Colvilles 26s. 9d.; and, awaiting the forthcoming dividend, Babcock & Wilcox rose to 76s. Dunlop Rubber at 77s. have also advanced strongly in anticipation of the dividend statement. Elsewhere, General Refractories rose afresh to 25s. 1½d. Helped by the Pinchin Johnson dividend and profit increase, paint shares were higher, where changed. Pinchin Johnson were 60s. 6d.; Lewis Berger £8½; and International Paint £7½. Similarly, textile shares have been helped by the increased J. & P. Coats payment, and Courtaulds at 55s. and British Celanese at 33s. 9d. have firmed up further on the removal of the rayon excise duty.

Further consideration of the results brought in by buyers for Associated Electrical Industries which have advanced to 84s. 6d.; as recently announced, Vickers has re-acquired a shareholding and thus revived its old association with this company. There has also been increased demand for General Electric, which rose to 97s.; and Crompton Parkinson were 33s. Cements were helped by the Tunnel Cement results and shares of the latter were 51s., while Associated Cement have risen to 69s. 3d., and elsewhere, British Plaster Board were 32s.

Triplex Glass 10s. ordinary units strengthened to 36s. 6d. and United Glass Bottle have been firm at 83s. 9d. on the financial results. The latter company appears to

have been a substantial E.P.T. payer in recent years, as has Sangers. The 5s. shares of the latter moved up to 35s. 9d. Elsewhere, Greiff-Chemicals 5s. ordinary were 14s. 6d., and Major & Co.'s 2s. ordinary 4s. 3d. Oils have been prominent with Anglo-Iranian at the new high level of £7 15/16, Shell £5½, Burmah Oil 83s. 9d., and Canadian Eagle advanced to 39s. 1½d. Awaiting the results there has been considerable activity in C. C. Wakefield shares, which, after touching 85s. 6d., encountered profit-taking and came back to 83s. 9d.

## British Chemical Prices

### Market Reports

**C**ONTINUED pressure for supplies characterised nearly all sections of the industrial chemicals market during the past week, and the call for contract deliveries was almost as great as prior to the fuel crisis. There have been no important price changes but the undertone remains strong. While the volume of overseas inquiry has continued on good scale, offers for export have been very much restricted by the home market. Similar conditions apply to the coal-tar products market, where there has been no outstanding feature.

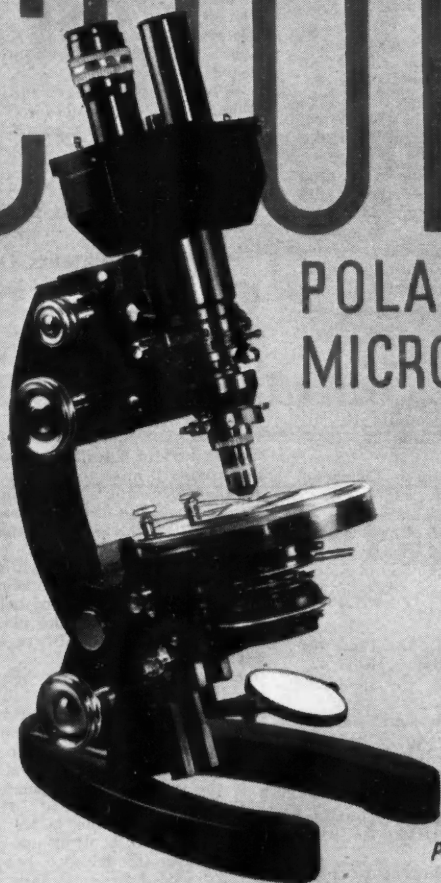
**MANCHESTER.**—An easing of the fuel supply situation is improving prospects for both production and consumption of heavy chemical products in the Lancashire area. On the Manchester market during the past week a strong undertone has been apparent throughout. Inquiry for caustic soda, soda ash and other alkali products has been active and persistent pressure for actual deliveries of these materials, as well as of a wide range of others, has been reported from home users. Shippers have also again been in the market, but they are not finding it easy to secure firm promises in respect of export business. Most classes of fertilisers are meeting with a steady demand and this is also reported in the tar products, the majority of which are moving promptly into consumption.

**GLASGOW.**—The Scottish heavy chemical trade has improved during the past week, supplies of many materials becoming easier. In the export trade inquiries have been as numerous as before, particularly for bleaching powder and precipitated chalk. Difficulties are being experienced in obtaining confirmation of orders due to high shipping freight rates, particularly for sulphuric acid. There have been a number of cases reported of overseas buyers being willing to pay f.o.b. prices, but stating that the freight rates from this country are too high, particularly in comparison with those from America.



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## Patents in the Chemical Industry

The following information is prepared from the Official Patents Journal. Printed copies of specifications accepted may be obtained from the Patent Office, Southampton Buildings, London, W.C.2., at 1s. each.

### Complete Specifications Open to Public Inspection

3 : 4-Di-(P-hydroxyphenyl)-hexanediol-3,4.—Roche Products, Ltd. Sept. 14, 1945. 22405/46.

Gas suitable for chemical syntheses.—Soc. Anon. Forni ed Impianti Industriali Ingg. de Bartolomeis. June 10, 1943. 4698/47.

Distillation furnaces.—Soc. Anon. Forni ed Impianti Industriali Ingg. de Bartolomeis. Aug. 29, 1940. 4699-4860/47.

Process for preparing halogen-containing high molecular substances.—Bataafsche Petroleum Maatschappij. Sept. 23, 1945. 34996/45.

Process for the decolourisation of articles produced from halogen-containing high molecular compounds.—Bataafsche Petroleum Maatschappij. Sept. 29, 1945. 35283/45.

Compounds having vitamin A activity.—Ortho Pharmaceutical Corporation. Sept. 20, 1945. 9438/46.

Directly obtaining non-hygroscopic ternary fertilisers.—Soc. de Produits Chimiques et Engrais D'Auby. June 29, 1945. 6178-79/47.

Lubricating oil compositions.—American Cyanamid Co. July 30, 1942. 14273/43.

Reaction of enol esters with anhydrides.—Carbide & Carbon Chemicals Corporation. Oct. 5, 1945. 27622/46.

Amines and quaternary ammonium salts.—Ciba, Ltd. Oct. 4, 1945. 28556-57/46. Monoazo-dyestuffs.—Ciba, Ltd. Oct. 4, 1945. 28558/46.

Polymeric materials.—E. I. Du Pont de Nemours & Co. Oct. 7, 1941. 13933/42.

Pesticidal compositions.—B. F. Goodrich Co. Oct. 1, 1945. 25069/46.

Beta-aroxy carboxylic acid compounds.—B. F. Goodrich Co. Oct. 5, 1945. 25945/46.

2-mercapto-4-keto-5, 6-dihydro 1, 3, 4-thiazine.—B. F. Goodrich Co. Oct. 5, 1945. 26121/46.

Beta-chloro and beta bromo carboxylic acids.—B. F. Goodrich Co. Oct. 5, 1945. 26122/46.

Beta substituted carboxylic acid compounds.—B. F. Goodrich Co. Oct. 5, 1945. 26579/46.

Beta-thio carboxylic acid compounds.—B. F. Goodrich Co. Oct. 5, 1945. 26580/46.

Beta (heterocyclic-thio) propionic acid.—B. F. Goodrich Co. Oct. 5, 1945. 26581/46.

Beta-dithiocarbamyl carboxylic compounds.—B. F. Goodrich Co. Oct. 5, 1945. 26582/46.

Polycarboxylic acids.—B. F. Goodrich Co. Oct. 5, 1945. 26821/46.

Beta-thio-carboxylic acid compounds.—B. F. Goodrich Co. Oct. 5, 1945. 26822/46.

Recovering ammonia from ammonia-containing gases.—Koppers Co., Inc. Oct. 4, 1945. 4531/46.

Regenerating bauxite.—N.V. De Bataafsche Petroleum Maatschappij. Oct. 4, 1945. 26493/46.

Selenium which is added by small quantities of substances for the purpose of increasing its conductivity.—N.V. Philips' Gloeilampenfabrieken. Oct. 1, 1945. 28911/46.

Treating selenium.—N.V. Philips' Gloeilampenfabrieken. May 24, 1944. 7053/47.

### Czech Patents Decree

CONSIDERABLE importance appears to be attached to a decree issued recently by the Czechoslovak authorities to the effect that all owners of Czechoslovak patents rights to which a material value had been attached on and after November 15, 1945, have to register with the appropriate authorities and make a declaration regarding the value of their rights. Based on this declaration, a levy of 5 to 30 per cent of the value declared will have to be paid not later than 45 days after the end of March, 1947. A failure to make this declaration may lead to a sequestration of the patent rights in question.

Although patents to which no material value is attached are exempt from this decree, it is, nevertheless, being feared that a sequestration may take place because the authorities may not always agree with the declaration of the patentee. The far-reaching effects of this decree become obvious if the fact is borne in mind that it does not merely apply to patent rights valid on the territory of the Czechoslovak Republic which, in some cases, form the basis for production under licence agreements, but also the import of goods made according to patented process abroad. However, it is admitted that in the latter case valuation may be most difficult if not impossible. It appears that this measure has been issued so as to raise revenue, but it is doubtful whether its long-term effects will be beneficial to the country's economy.

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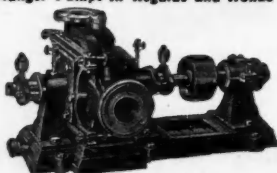
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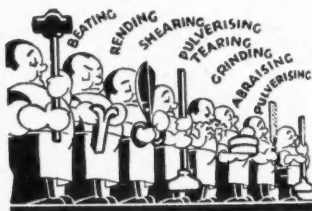
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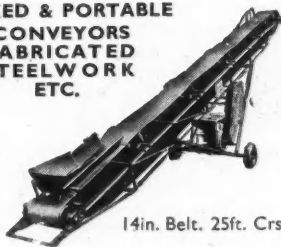
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